

# ANALYTICAL ABSTRACTS

R.R.

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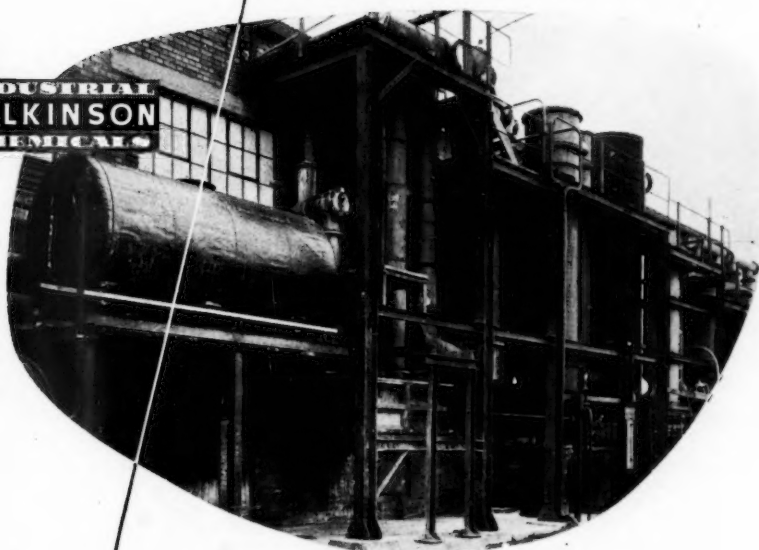
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## ANALYTICAL ABSTRACTS

## 1.—GENERAL ANALYTICAL CHEMISTRY

219. The application of thio salts in analysis. III. A new scheme of qualitative analysis. Parts A and B. I. K. Taimni and R. P. Agarwal (*Anal. Chim. Acta*, 1953, 9 [3], 208-215; 216-222).—A. A conc solution of  $\text{NH}_4$  sulphide is prepared by saturating aq.  $\text{NH}_3$  with  $\text{H}_2\text{S}$ , and is protected from oxidation. The use of this reagent in place of  $\text{H}_2\text{S}$  affords quicker and more complete pptn. of sulphides from acid solution; it also leads to a more satisfactory separation of the Cu group from the As group than is possible with yellow  $\text{NH}_4$  sulphide. For group separation the treatment is divided into 7 procedures, as follows. (i) Evaporate with conc.  $\text{HNO}_3$ , then with conc.  $\text{HCl}$ , and extract the residue with dil.  $\text{HCl}$  adding aq. Br to oxidise Sn and Tl. The following remain undissolved;  $\text{AgCl}$ ,  $\text{AgBr}$ ,  $\text{AgI}$ ,  $\text{BaSO}_4$ ,  $\text{SrSO}_4$ ,  $\text{PbSO}_4$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{SnO}_2$ ,  $\text{Sb}_2\text{O}_3$ ,  $\text{HgS}$ ,  $\text{SnS}_2$ ,  $\text{CaF}_2$ ,  $\text{SiO}_2$ ,  $\text{WO}_3$ ,  $\text{TiO}_2$ ,  $\text{Zr(OH)PO}_4$  and complex ferrocyanides. (ii) Treat the solution from (i) with  $\text{NH}_4$  sulphide until strongly alkaline, filter, extract the ppt. with  $\text{NH}_4$  sulphide and with hot water and combine the filtrates. (iii) Make the filtrate from (ii) 6N in  $\text{HCl}$ , boil and filter. The ppt. contains the sulphides of As, Se, Te, Mo, Pt, Au and V. (iv) Treat the filtrate from (iii) with  $\text{NH}_4$  sulphide in excess, then with dil.  $\text{HCl}$ , and filter. The ppt. consists of sulphides of Sb, Sn (and V); the filtrate contains chlorides of alkaline earth metals and alkalis. (v) Treat the ppt. from (ii) with  $\text{NH}_4$  sulphide for 2-3 min. and filter. The insoluble portion contains Hg, Pb, Bi, Cu, Cd, Ni, Co and Tl. (vi) Treat the filtrate from (v) with  $\text{NaOH}$ , boil, and filter. The filtrate contains Cr, U, Zn, Be and Al. (vii) Dissolve the ppt. from (vi) in the min. amount of conc.  $\text{HCl}$  and dilute to an acidity of about 0.5N; add an excess of oxalic acid. Oxalates of Ce and Th are precipitated; the filtrate contains Mn, Fe, Ti, Zr, (Tl) and alkaline earths.

B. A detailed scheme, based in part on the Noyes and Bray scheme, is given for the separation of the members of each group in the foregoing system.

W. C. JOHNSON

220. The application of thio salts in analysis. II. Estimations based on decomposition of thio salts. Part B. Estimation of molybdenum and tin. I. K. Taimni and R. P. Agarwal (*Anal. Chim. Acta*, 1953, 9 [3], 203-207).—Molybdenum in solution as  $\text{MoO}_4^{2-}$  is treated with an excess of 15 N  $\text{NH}_4$  sulphide or 2 N Na sulphide soln. and then with dil.  $\text{HCl}$  until the colour of the thio salt is discharged. The mixture is boiled and the pptd. sulphide is allowed to settle; the sulphide is then filtered, washed with water, alcohol and ether, dried in a vac. desiccator and weighed as  $\text{MoS}_2 \cdot 2\text{H}_2\text{O}$ . Tin in solution as  $\text{SnCl}_4$  is treated with aq.  $\text{NH}_3$  until a white precipitate of  $\text{Sn(OH)}_4$  appears, and is then treated with a large excess of 2 N Na sulphide soln. The yellow soln. of Na thiostannate is decomposed with an excess of

2-3 N acetic acid and set aside for 10 min.; the precipitate is filtered, washed with water, alcohol and ether, dried in a vac. desiccator and weighed as  $\text{SnS}_2 \cdot 2\text{H}_2\text{O}$ .

W. C. JOHNSON

221. Titration of acids and bases in non-aqueous solutions. I. H. Ballczo (*Mitt. Chem. ForschInst. Ind.*, 1953, 7, 104-112).—A review with 45 references covering theoretical and practical considerations.

P. S. ARUP

222. Catalysis and induction with redox-titrations. VI. The law of chemical induction. A. Schleicher (*Z. anal. Chem.*, 1953, 139 [5], 321-326).—By analogy with electrical induction, the law of multiple proportions in its application to redox reactions is described as the law of chemical induction. The disproportionation of  $\text{Cu}^I$  and of  $\text{Fe}^{III}$  occurs by self-induction, the magnitude of which may be found by means of the activity coeff. The analogy is extended to heterogeneous catalysis, as in the example of oxyhydrogen gas in contact with Pt.

R. E. STANTON

223. Mineralisation of organic material in the Kjeldahl method. I. Ribas and D. Vázquez-Gesto (*Inf. Quim. Anal.*, 1953, 7 [2], 29-42).—A method particularly suitable for N-ring compounds consists in treating 10 mg of the test substance in 2 ml conc.  $\text{H}_2\text{SO}_4$  with a catalytic mixture composed of 3 g  $\text{K}_2\text{SO}_4$ , 0.05 g  $\text{HgSO}_4$  and 1 ml of a 0.5 per cent. soln. of Se in conc.  $\text{H}_2\text{SO}_4$ . The following may be rapidly analysed in this way: tryptophan, nicotinic acid, histidine hydrochloride, sparteine perchlorate, cystine tartrate, cinchonine hydrochloride, atropine sulphate,  $\alpha\beta$ -dipiperidyl hydrochloride, glycine, alanine, aspartic acid, creatinine and tyrosine.

L. A. O'NEILL

224. Determining only one compound in a mixture. Short spectrophotometric method. E. Allen and W. Rieman III (*Anal. Chem.*, 1953, 25 [9], 1325-1331).—The problem of determining one component in a multicomponent system is discussed in the light of a new mathematical treatment. Provided certain relations hold good, the determination can be made by taking absorbance readings at only two or three wavelengths no matter how many compounds are present. The well-known base-line method is a special case of the method presented. The validity of the two-wavelength method has been verified experimentally by the determination of pyridine in a six-component system of related compounds.

J. M. JACOBS

225. Comparative characteristics of some ion-exchange materials. II. D. I. Ryabchikov, M. M. Senyavin and K. V. Filippova (*J. Anal. Chem., U.S.S.R.*, 1953, 8 [4], 220-224).—Earlier data (*Brit. Abstr. C*, 1953, 44) are supplemented by results of experiments on swelling capacity and sp.gr. of air-dried and oven-dried ion-exchange substances, and on their exchange capacities at various pH values.

G. S. SMITH

**226. Chromatographic analysis.** D. I. Ryabchikov and M. M. Senyavin (*J. Anal. Chem., U.S.S.R.*, 1953, **8** [4], 195-210).—A review with 96 references. G. S. SMITH

**227. Chromatographic method in qualitative analysis.** I. K. M. Olshanova and K. V. Chmutov (*J. Anal. Chem., U.S.S.R.*, 1953, **8** [4], 211-216).—The orders of adsorption of cations and anions on various adsorbents (alumina, Permutit, kaolin, synthetic resins, etc.) are studied for each group of ions. Results are tabulated, and recommendations regarding most suitable types of adsorbents for particular kinds of analytical separations in chromatographic qual. analysis are given. G. S. SMITH

**228. A simple method for further separating mixtures of substances after paper chromatography.** K. Schlögl and A. Siegel (*Hoppe-Seyl. Z.*, 1953, **292**, 263-268).—A mixture of substances with similar  $R_F$  values is frequently contained in one large spot. This can be transferred to a fresh chromatogram by cutting out the dried spot and placing it on the new paper. It is held in place between two plates of glass. It is often more convenient to use this technique than to use two-dimensional chromatography or elution. Practical examples of the method are given for amino-acids, azo dyes and alkylphenoxycetic acids. L. ROWE

**229. An improved method for gradient elution chromatography.** T. K. Lakshmanan and S. Lieberman (*Arch. Biochem. Biophys.*, 1953, **45** [1], 235-237).—A method for gradient elution is described; it is based on rates of flow,  $R_1$  and  $R_2$ , into and out of a mixing chamber into the column, the rates being independently variable. A Hershberg dropping funnel (*cf. Ind. Eng. Chem., Anal. Ed.*, 1936, **8**, 313) connected to a mixing chamber having a magnetic stirrer is a suitable apparatus. If  $V_0$  is the initial vol. of pure solvent in the mixing chamber, and  $C_0$  the solvent concn. in the reservoir, then  $C = C_0 \{1 - [a/(a + bt)]^{1/2}\}$ , where  $a = V_0/R_1$  and  $b = 1 - (R_2/R_1)$ . Appropriate values of  $a$  and  $b$  will give a linear, convex, or concave gradient of the concn. - time curve as required, so giving flexible chromatographic analysis of general applicability. A curve is given of the resolution of a mixture of five 17-ketosteroids in a concave elution chromatogram. H. H. HODGSON

**230. A critical review of the  $R_F$  value in paper chromatography.** G. Zimmerman (*Z. anal. Chem.*, 1953, **138** [5], 321-332).—In the literature many conflicting statements concerning the accuracy of the  $R_F$  value are found. The following factors have been found to be decisive in the determination of the  $R_F$  value: the development phase, quality of the paper, the fibre arrangement of the paper, the nature of the development, the distance travelled for development, the distance of the development phase reservoir from the starting line, the concn. of the substance, other substances present and the temp. R. J. MAGEE

**231. Use of precipitation chromatography in qualitative semi-microanalysis.** A. I. Komlev and L. I. Tsimbalista (*J. Anal. Chem., U.S.S.R.*, 1953, **8** [4], 217-219).—For semi-micro inorganic qual. chromatography a column in a glass tube 4-5 cm long and 3-4 mm internal diameter is used. The

column is prepared either dry or wet. With the dry column the carrier adsorbent, e.g.,  $Al_2O_3$  or a synthetic resin, well ground with the precipitant is packed into the tube to give a uniform density (by dropping the column 5-10 times through a height of 50 cm). With the wet column the mixed charge is washed with a solution of the precipitant before use. The weight of the charge is 0.1-0.2 g and the length of the working part is  $\approx 1$  cm. To detect  $Cl^-$ ,  $Br^-$ , and  $I^-$  in a mixture, a dry column of  $Al_2O_3$  containing 1-10 per cent. of  $AgNO_3$  or  $Ag_2SO_4$  is used and 3 drops of the halide soln. (0.0001-1.0 N) are passed through the column. Separate zones whose length depend on the concn. (2 mm with 0.1 N) appear after 5-15 min. in sunlight. Partial or indefinite indications are given by the passage of adsorption indicators through the column. The min. amounts detectable and the limiting concn. are:  $I^-$ , 8  $\mu g$ , 1:7000;  $Cl^-$  and  $Br^-$ , 14-15  $\mu g$ , 1:4000. To detect the cations of the Ag, Hg, and Pb group a moist column of  $Al_2O_3$  and KI is necessary. Separate zones of  $AgI$ ,  $Hg_2I_2$ , and  $PbI_2$  appear. With the Cu group,  $Al_2O_3$  moistened with saturated  $Na_2S$  or 2 N  $(NH_4)_2S$  solution is used. G. S. SMITH

**232. Reduction of liquid flow in paper electrophoresis.** M. A. Jermyn and R. Thomas (*Nature*, 1953, **172**, 728-729).—Movement of spots of trinitrobenzene and *o*-nitroaniline was used for following liquid flow. At and below pH 5.0 trinitrobenzene is uncharged and its position is determined by spraying with 5 per cent. dimethylaniline in ethanol which produces a pink colour that is intensified by exposure to  $NH_3$  gas. At pH 7.0 and above, *o*-nitroaniline is uncharged and forms a yellow spot. By applying the spots before wetting the paper and allowing most of the indicator solvent to evaporate, then saturating the strip with buffer and, without blotting, immediately clamping it between glass plates, no appreciable spreading of the spots occurred. Moreover, by sealing the plates along the edges and binding them transversely with adhesive cellulose tape, evaporation was reduced and fairly uniform flow was observed throughout the length of the sealed region of the strip when the current was passed. The use of buffer-saturated paper completely arrested inward flow from the ends and replaced it by a very slight outward flow caused by evaporation of the buffer from the exposed ends of the strip. When proteins were applied to the paper before saturation with buffer, they were adsorbed more strongly than were the indicators. This effect was avoided, without alteration of the liquid flow, by spotting the protein solution on the buffer-saturated strip at a locally blotted site. The effect of electro-osmosis on the liquid flow is discussed and means are described for counteracting this effect. B. VINEY

**233. Indirect polarographic analysis by means of complex components with one anodic depolarisation step.** D. Lydersen (*Z. anal. Chem.*, 1953, **139** [5], 327-339).—A method of indirect polarographic analysis by means of complex compounds is discussed. The method depends on the accurate measurement of the height of the depolarisation step. The practicability of the method and the optimum conditions for accurate results are discussed, data from the literature on the determination of Ca, Mg, and  $Fe^{III}$  in presence of ethylenediaminetetra-acetate being used. R. E. STANTON

234. Empirical order of solubilities of ferrocyanides. R. V. Vorontsov (*J. Anal. Chem., U.S.S.R.*, 1953, 8 [4], 228-230).—It was assumed that a cation having a less sol. ferrocyanide should replace the cation in the ppt. of a more sol. ferrocyanide. Solubilities of ferrocyanides in approx. neutral solution were assessed on this assumption by four different methods of studying the interactions. All results were consistent and gave the following order of cations with respect to decreasing solubilities of their ferrocyanides:  $Pb^{++}$ ,  $Al^{+++}$ ,  $Bi^{+++}$ ,  $Sb^{+++}$ ,  $Mn^{++}$ ,  $Cd^{++}$ ,  $Co^{++}$ ,  $Ni^{++}$ ,  $Fe^{++}$ ,  $Zn^{++}$ ,  $Fe^{+++}$ ,  $Cu^{++}$ ,  $Ag^{+}$ . The results provide methods for purification of Ni electrolytes, detection of Cd in presence of Cu, and detection of Al in presence of all other cations.

G. S. SMITH

## 2.—INORGANIC ANALYSIS

235. Preparation of water samples for deuterium analysis in the mass spectrometer. F. P. Chinard and T. Enns (*Anal. Chem.*, 1953, 25 [9], 1413-1414).—The reduction of the separate water samples by means of the conventional zinc trains, with transfer of the gas into a sample tube by means of a Toepler pump, that is so tedious, may be avoided and a considerable reduction may be made in the vol. and surface of the apparatus by heating the water samples in presence of Zn in individual sample tubes; the tubes are described. The sample of water should be  $> 0.01$  ml as a pressure of  $\approx 24$  atm. would be developed by 0.1 ml of water in a 10-ml sample tube.

J. M. JACOBS

236. Estimating the tritium content of tritiated water. Wilmer A. Jenkins (*Anal. Chem.*, 1953, 25 [10], 1477-1480).—A method is described for determining the tritium content of tritiated water with an accuracy of 15 per cent. The activity of solid  $NH_4Cl$  which had been rendered radioactive by exchange with tritiated water is measured in a windowless flow counter. The application of the method to other exchange and tracer experiments is described.

O. M. WHITTON

237. Paper chromatography of alkali sulphates and chlorides. R. Viswanathan (*J. Sci. Ind. Res. India, B*, 1953, 12, 331-332).—Alkali sulphates and chlorides are separated chromatographically with  $C_2H_5OH$  as developing solvent. No separation of the sulphates of Na and K ( $R_F$  values both zero) is observed, but NaCl, KCl and  $NH_4Cl$  ( $R_F$  values 0.20, 0.10 and 0.33, respectively) can be separated.

D. BAILEY

238. Gravimetric determination of lithium by precipitation as trilithium phosphate. E. R. Caley and G. A. Simmons, jun. (*Anal. Chem.*, 1953, 25 [9], 1386-1389).—To the solution of the alkali metals as chlorides or sulphates  $\approx 2M$  choline phosphate is added and the solution is heated on a steam bath. One hr. after the first appearance of a ppt. an equal vol. of propan-2-ol is added; the solution is set aside for 2 hr. and filtered hot. The ppt. is washed with successive small portions ( $\approx 40$  ml in all) of a saturated soln. of  $Li_3PO_4$  in 50 per cent. propan-2-ol, and ignited for 30 min. at a low red heat. If  $> 10$  mg of Na is suspected in the original solution, the ppt. is dissolved in 2 ml of 4M HCl and reprecipitated. By this method Li can be determined accurately in presence of Na and K. Sulphate ions do not interfere. About 2 mg is the smallest amount of Li that can be determined by this procedure.

J. M. JACOBS

239. Micro-determination of potassium with radioactive cobaltinitrite. E. Sánchez Serrano and I. López Santos (*Inf. Quim. Anal.*, 1953, 7 [2], 43-50).—Potassium is pptd. from aq. alcoholic media with Na Ag cobaltinitrite containing  $^{60}Co$  and the activity of the K Ag cobaltinitrite ppt. is measured by a Geiger counter. Microgram amounts can be determined. Application of the method to blood analysis is described.

L. A. O'NEILL

240. Determination of rubidium and caesium based on the difference in stability of their tri-iodides. H. Yamatera (*J. Inst. Polyt. Osaka*, 1953, 4 [1], 53-58).—Determination of Rb and Cs together in the presence of K and of Cs in the presence of K and Rb is carried out by titration of liberated I formed by the dissociation of the tri-iodides;  $CCl_4$  containing I is used as solvent. Experimental conditions are limited and some anomalies appear, suggesting the formation of double tri-iodides of Cs and Rb, but this is unconfirmed.

G. R. WHALLEY

241. Determination of copper as cuprous thiocyanate. E. Kurzyniec and A. Kulpiński (*Prace Kom. Nauk. Farm. Polsk. Acad.*, 1952, 4, 33-42).—The method for determining Cu in form of CuCNS (first published by Rivot, *Compt. Rend.*, 1854, 38, 868) and modifications introduced by other authors are reviewed. By varying the experimental conditions, the following modifications were found to give accurate results. The copper salt solution, provided that it does not contain strong free acids, is treated with sulphurous acid; Cu is precipitated with ammonium thiocyanate on stirring. The precipitate is allowed to settle for 20 min., filtered through a dried and weighed Gooch crucible, washed 2-3 times first with water containing a small amount of ammonium thiocyanate or  $H_2SO_4$ , then with pure water and finally with a (1+1) alcohol-ether mixture, and dried for 10 min. at  $120^\circ C$ .

A. STORFER

242. The micro-estimation of copper with triphenylmethylarsonium thiocyanate. K. W. Ellis and N. A. Gibson (*Anal. Chim. Acta*, 1953, 9 [4], 368-373).—Ten ml of Cu solution ( $10-100 \mu g/ml$ ) at pH 1-5 are added to 5 ml o-dichlorobenzene in a small separating funnel and, if  $NO_3^-$  or  $Cl^-$  are present in large amount, 1 ml of N sodium acetate is added. One per cent.  $(C_6H_5)_3CH_2AsCl$  (2 ml) is added and then (reversing the order results in loss as CuCNS) 1 ml of 25 per cent.  $NH_4CNS$ . The  $[(C_6H_5)_3CH_2As]_2[Cu(CNS)_4]$  (also prepared as a solid by pptn. in aq. medium) dissolves on shaking in the org. layer; the aq. layer is twice extracted with o-dichlorobenzene. The combined filtered extracts are made up to 25 ml and the absorption is measured on a Spekker absorptiometer (filter, blue-green 603).  $F^-$ , acetate and  $SO_4^{--}$  do not interfere except in large amounts and the interference of  $Cl^-$  and  $NO_3^-$  is eliminated by buffering to pH  $> 3$ .  $PO_4^{---}$  interferes. Interference of  $Cr^{+++}$  and  $Fe^{+++}$  is sufficiently slight for a partial separation to suffice.  $Mo^V$  interferes but can be oxidised to  $Mo^{VI}$ . Co interferes strongly.

E. J. H. BIRCH

243. Spectrophotometric determination of copper with cuproine [2:2'-diquinolyl]. J. Hoste, J. Eeckhout and J. Gillis (*Anal. Chim. Acta*, 1953, 9 [3], 263-274).—The effect is determined of a number of possible influences on the following general method. A solution of  $125 \mu g$  of Cu as  $CuSO_4$  in 25 ml of water is treated with some crystals of  $NH_4OH.HCl$  and is shaken with 5 ml of freshly distilled isopentyl alcohol containing 0.02 per cent.

of cuproine. The  $\text{Cu}^I$  forms a purple complex in the organic layer, which is decanted. The extraction is repeated until the  $\text{Cu}^I$  is completely removed from the aq. layer, the extracts are combined, diluted to 25 ml, and the extinction is determined in a Beckman DU spectrophotometer. The max. is at 546  $\text{m}\mu$ ;  $\epsilon = 6430$ . The partition coeff. of isopentyl alcohol to water is 1680. The min. shaking time is 50 sec. for a water to isopentyl alcohol ratio of 1:1 and 2 min. for a ratio of 10:1. Extraction is complete within the pH range 2-9. The following do not interfere: Al, As, Ba, Ca, Cd, Co,  $\text{Fe}^{III}$ ,  $\text{Fe}^{II}$ , Li, Mg,  $\text{Mo}^{VI}$ ,  $\text{Mn}^{II}$ ,  $\text{NH}_4$ , Ni,  $\text{Sb}^{III}$ ,  $\text{Sn}^{II}$ , Sr,  $\text{Ti}^{IV}$ , VV,  $\text{W}^{VI}$ , Zn. Of the common anions only cyanide, thiocyanate and oxalate interfere. Variation of reagent concn. does not markedly affect the extinction. Beer's law is followed. Because of the favourable partition coeff., 99 per cent. of the  $\text{Cu}^I$  can be extracted from 50 ml with only one 5-ml portion of the reagent soln. and this is the basis of an empirical alternative method that has the advantages of greater sensitivity and rapidity. The mean square error of both methods is  $< 1$  per cent. Details are given for the application of these methods to the determination of Cu in plants, water, animal tissues and blood plasma, lamp black, and alloy steels.

W. C. JOHNSON

**244. Determination of copper in metallurgical analysis. Use of 2:2'-biquinoline (2:2'-diquinolyl).** R. J. Guest (*Anal. Chem.*, 1953, 25 [10], 1484-1486).—In the simple spectrophotometric method described for determining Cu, a coloured complex is formed by 2:2'-diquinolyl (cuproine) and  $\text{Cu}^I$ , and extracted by *n*-pentyl alcohol from a weakly acid soln. Samples containing between 0.001 and 10 per cent. or even more Cu can be analysed. Results are reproducible under routine conditions. After the sample has been dissolved, a single determination takes less than 15 min.

O. M. WHITTON

**245. Simultaneous electro-deposition of copper and lead from nitric acid—hydrofluoric acid—beryllium nitrate solution.** C. Goldberg (*Anal. Chem.*, 1953, 25 [9], 1405).—Quantitative results for simultaneous electro-deposition of Cu and  $\text{PbO}_2$  from  $\text{HNO}_3$ ,  $\text{HNO}_3$ -HF, and  $\text{HNO}_3$ -HF- $\text{BeF}_2$  solutions are compared, and the results with the last of these solutions are compared with those by standard analytical procedures.

D. A. PANTONY

**246. The volumetric determination of silver in lead.** G. Mannelli (*Anal. Chim. Acta*, 1953, 9 [3], 232-234).—One gram of Pb is dissolved in  $\text{HNO}_3$  and the soln. is evaporated to dryness. The residue is dissolved in a few ml of water, 2-5 ml of 30 per cent. aq.  $\text{NH}_4$  acetate are added and the soln. containing 2-3 drops of 0.1 per cent. aq. bromophenol blue as adsorption indicator is titrated with 0.02 N KI. In acid soln. the colour change is from grey-purple to green-grey with a return to grey-purple at the end-point; in less acid solutions the change is from violet to purple. Quantities of Ag down to 0.1 mg can be determined and the Ag:Pb ratio can be as low as 1:14,000. W. C. JOHNSON

**247. Solubility of silver pyrophosphate in alkali pyrophosphates.** M. Kohn (*Anal. Chim. Acta*, 1953, 9 [3], 229-231).—One hundred ml of cold saturated aq.  $\text{Na}_4\text{P}_2\text{O}_7$  dissolve about 0.0324 g of  $\text{Ag}_2\text{P}_2\text{O}_7$ . Aqueous  $\text{K}_4\text{P}_2\text{O}_7$  of molar strength equal to that of saturated aq.  $\text{Na}_4\text{P}_2\text{O}_7$  dissolves about twice as much  $\text{Ag}_2\text{P}_2\text{O}_7$ , and the solubility increases with increasing concn. of  $\text{K}_4\text{P}_2\text{O}_7$ . W. C. JOHNSON

**248. Analytical chemistry of fluoride complex compounds (supplemental reports).** T. Sawaya (*Technol. Rep. Tôhoku Univ.*, 1952, 17 [1], 43-57).—Direct and indirect methods for the determination of beryllium by means of the fluoride complex compound are described. The determination of beryllium and aluminium in a mixture without previous separation is also given. Uranium is determined by acidimetric titration by means of the reaction of the fluoride complex compound; several indicators are used. P. N. R. NICHOLS

**249. Colorimetric determination of calcium with ammonium purpurate.** Max B. Williams and J. H. Moser (*Anal. Chem.*, 1953, 25 [9], 1414-1417).—The method is based on the formation by murexide of a coloured complex with  $\text{Ca}^{++}$  in aq. solution (*cf.* Schwarzenbach *et al.*, *Helv. Chim. Acta*, 1949, 32, 1314). The absorbancy is measured at 506  $\text{m}\mu$  in solutions of which the pH has been adjusted to 11.3 with NaOH. The max. concn. in mg per litre of certain ions which cause no change in the slope of the working curve are: Na, 300; K, 500; Cs, 500; Mg, 5; Sr, 1; and Ba, 5. J. M. JACOBS

**250. The volumetric determination of calcium and magnesium.** R. L. Stephens (*J. Pharm. Pharmacol.*, 1953, 5 [10], 709-714).—The method entails bringing the Ca and Mg salts into soln. with a min. quantity of HCl with 150 ml water and adding excess of 0.5 N di-sodium ethylenediaminetetra-acetate. The soln. is then titrated with 0.5 N  $\text{CaCl}_2$  in one of two ways. In the first method the indicator is naphthol green and murexide ground in NaCl. In the second method, for combined Ca and Mg, the indicator is Eriochrome Black T ground with NaCl, and an excess of ammonia is added. These indicators give good end-points. Some of the difficulties are described, *e.g.*, murexide is not suitable in the presence of  $\text{PO}_4^{3-}$  or in the presence of Fe. The second method is satisfactory in pH range 9-6-10.4, and the first above pH 12.

E. H. JOHNSON

**251. Complexometric determination of calcium and magnesium in media very rich in chlorides (crude sea salt and brines).** A. de Sousa (*Anal. Chim. Acta*, 1953, 9 [4], 305-308).—Crude sea salt (10 g) is dissolved in 200 ml  $\text{H}_2\text{O}$ , the pH is adjusted to  $> 12$  with NaOH and the solution is titrated with di-sodium ethylenediaminetetra-acetate and murexide (saturated aq. solution  $< 2$  weeks old) indicator to give the titre of  $\text{Ca}^{++}$  alone.  $\text{Al}^{+++}$  and  $\text{Fe}^{+++}$  (not usually found in sea salt) interfere. Titre of Mg is found after buffering with aq.  $\text{NH}_3$ - $\text{NH}_4\text{Cl}$ , adding 0.4 per cent. Eriochrome Black T—ethanol as indicator and titrating similarly at 40°-50° C. The accuracy is better than 0.5 per cent.

E. J. H. BIRCH

**252. The analytical use of trithiocarbonic acid: the determination of zinc.** W. Pilz (*Monatsh. Chem.*, 1952, 83, 471-477).—A procedure is outlined for the determination of zinc by trithiocarbonic acid. Suitable conditions of pH for the precipitation are more easily attained than by the use of  $\text{H}_2\text{S}$ . Only in the pH range 5.5-8.0 is any marked retardation of precipitation encountered. Outside this pH range the longest period of induction is 3 min. From acid solution the  $\text{ZnS}$  is crystalline and easily filtered. From alkaline solution it is flocculent and often slimy. For quant. precipitation a 4-5 per cent. excess of trithiocarbonic acid is necessary but in practice an excess up to 30 per cent. will cause no

trouble. The zinc soln. may contain up to 0.28 g in 100 ml. Precipitation may be carried out in HCl,  $\text{H}_2\text{SO}_4$ , acetic acid or alkaline soln. The maximum normality of the filtrate from the precipitation, permissible is: HCl, 0.02 N;  $\text{H}_2\text{SO}_4$ , 0.02 N; acetic acid, 2.0 N. In alkaline soln. an excess of NaOH or aq.  $\text{NH}_3$  causes trouble. The precipitate is dissolved in dil.  $\text{H}_2\text{SO}_4$  and titrated with  $\text{K}_4[\text{Fe}(\text{CN})_6]$ . Addition of bromine water and boiling for 5 min. eliminates false end-points caused by the presence of the lower oxides of sulphur.

R. J. MAGEE

253. The specific titration of zinc and cadmium with complexones in the presence of other metal ions. H. Flaschka (*Z. anal. Chem.*, 1953, **138** [5], 332-337).—Zinc and cadmium in the presence of Co, Ni, Cu and Hg can be determined accurately by titration with 0.1 N di-sodium ethylenediaminetetra-acetate dihydrate (Complexone III). With KCN all the metals are converted into complex cyanides, but on addition of formaldehyde decomposition of the cyanides of Zn and Cd only occurs. It is possible to determine Ca and Mg or Pb complexometrically in the same sample. Interference from other metals and the possibility of the use of the procedure for the analysis of alloys is discussed.

R. J. MAGEE

254. Coulometric determinations of submicrogram amounts of cadmium and zinc with stationary, mercury-plated platinum electrodes. K. W. Gardiner and L. B. Rogers (*Anal. Chem.*, 1953, **25** [9], 1393-1397).—The coulometric determination of  $\mu\text{g}$ -quantities of reducible metals by electrolytic deposition on Hg, followed by quant. soln., has been extended to mercury-plated platinum electrodes, which have the same favourable hydrogen overvoltage. The precision attainable with these electrodes under optimum experimental conditions is good; when 5 successive deposition (5 min.) and solution (40 sec.) cycles are made at  $10^{-3}$  M and  $10^{-5}$  M concn. of Cd, mean values of  $1.21 \times 10^{-7}$  and  $1.35 \times 10^{-8}$  g of Cd were recovered, respectively, with coeff. of variation of 3.0 and 6.7 per cent. For Zn the soln. peak is much broader and a much longer period of conditioning of the electrode is required. In order to obtain a more adherent coating of Hg so that the electrode can be rotated, Ag was substituted for Pt, but the erratic behaviour of the resulting electrodes render their application to micro-coulometric measurements of limited value.

J. M. JACOBS

255. Alkalimetric determination of mercury and its separation from other heavy metals. Mihir Nath Das (*Anal. Chem.*, 1953, **25** [9], 1406-1408).— $\text{Hg}^{2+}$  salts can be titrated directly with NaOH by means of phenolphthalein indicator in  $\approx 10$  per cent. aq. acetone as the titration medium; the  $\text{HgO}$  formed during the titration remains in solution, probably owing to formation of  $3\text{HgO} \cdot 2\text{C}_2\text{H}_5\text{O}$ . Difficulties that arise in direct titration with NaOH are overcome by treating the complex with KI or  $\text{Na}_2\text{S}_2\text{O}_3$  and titrating the liberated alkali (two equiv. per mol. of Hg) with standard acid. Cu, Ni and Co are pptd. by addition of excess NaOH and are filtered off before the above procedure is followed. The method works satisfactorily in presence of  $\text{Cl}^-$ ,  $\text{SO}_4^{2-}$ ,  $\text{NO}_3^-$ ,  $\text{ClO}_4^-$ , and acetate, but  $\text{Br}^-$ ,  $\text{I}^-$ ,  $\text{CNS}^-$ , and  $\text{S}_2\text{O}_3^{2-}$  interfere seriously, while  $\text{CN}^-$  and  $\text{PO}_4^{3-}$  interfere with the phenolphthalein colour change.

J. M. JACOBS

256. Enhancement of the reducing properties of metallic mercury in presence of complex-forming ions. F. Burriel-Marti, F. Lucena-Conde and S. Bolle-Tacchco (*Anal. Chim. Acta*, 1953, **9** [4], 293-304).—In presence of  $\text{CNS}^-$  metallic Hg reduces  $\text{Fe}^{3+}$  to  $\text{Fe}^{2+}$ ,  $\text{Cu}^{2+}$  to  $\text{CuCNS}$ ,  $\text{VO}_4^{3-}$  and  $\text{MoO}_4^{2-}$  in acid medium to V and Mo complex thiocyanates, and  $\text{Fe}(\text{CN})_6^{3-}$  to  $\text{Fe}(\text{CN})_6^{4-}$  in acid, alkaline and neutral media, but  $\text{Ti}^{3+}$  and  $\text{Cr}^{3+}$  are not reduced. In presence of  $\text{CN}^-$ , Hg reduces  $\text{O}_2$  to  $\text{H}_2\text{O}_2$ ,  $\text{Fe}(\text{CN})_6^{3-}$  to  $\text{Fe}(\text{CN})_6^{4-}$  and  $\text{MnO}_2$  to  $\text{Mn}(\text{CN})_6^{4-}$ , but  $\text{CrO}_4^{2-}$ ,  $\text{VO}_4^{3-}$ ,  $\text{MoO}_4^{2-}$ ,  $\text{IO}_3^-$ ,  $\text{BrO}_3^-$ ,  $\text{ClO}_3^-$  are not reduced. For the estimation of  $\text{Fe}^{3+}$  the solution ( $< 70$  mg  $\text{Fe}/100$  ml) is acidified (5 ml conc.  $\text{H}_2\text{SO}_4$ ) and 5 ml  $\text{M KCN}$  and 10 ml Hg are added and the mixture is stirred until the solution is decolorised ( $\approx 30$  sec.). The Hg is separated by decantation and washing, or by addition of  $\text{CCl}_4$  to form a layer between it and the solution; after addition of  $\text{H}_3\text{PO}_4$  and 5 ml of  $\text{M HgSO}_4$  (to mask the  $\text{CNS}^-$ ) the solution is titrated with  $\text{K}_2\text{Cr}_2\text{O}_7$  or  $\text{Ce}(\text{SO}_4)_2$ . HCl does not interfere at concn.  $< 0.1$  M.  $\text{Mn}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Ti}^{3+}$ ,  $\text{Cr}^{3+}$ ,  $\text{Cd}^{2+}$ ,  $\text{Al}^{3+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Co}^{2+}$  and  $\text{Bi}^{3+}$  do not interfere, but  $\text{MoO}_4^{2-}$ ,  $\text{VO}_4^{3-}$ ,  $\text{Cu}^{2+}$  and  $\text{NO}_3^-$  do. For estimation of  $\text{Fe}(\text{CN})_6^{3-}$ , 50 ml of the solution are stirred with 0.1 M KCN and Hg until decolorised and then heated in alkaline solution to decompose  $\text{H}_2\text{O}_2$ ; after acidification the  $\text{Fe}(\text{CN})_6^{4-}$  is titrated with  $\text{Ce}(\text{SO}_4)_2$ ; a small empirical correction is necessary.

E. J. H. BIRCH

257. Separation of radium D, E and F by paper chromatography. E. E. Dickey (*J. Chem. Educ.*, 1953, **30** [10], 525).—A standard method (Rao, P.S., and Beri, R. M., *Brit. Abstr. C*, 1952, 148) is used with lead (Ra-D) and Bi (Ra-E) carriers.

N. PILFEL

258. Determination of very small amounts of indium. R. Bock and K.-G. Hackstein (*Z. anal. Chem.*, 1953, **138** [5], 337-339).—A procedure is outlined for the preparation of a series of standard solutions from which small amounts of indium (lower limit 0.1  $\mu\text{g}$ ) can be determined by the fluorescence of the oxyquinolate in chloroform solution. Elements such as Al whose hydroxyquinolates can be extracted with chloroform must be absent or removed.

R. J. MAGEE

259. Studies on the separation of the rare earths and their accompanying elements. Part I. By using the hydrolysis of a buffered solution. Z. Hagiwara (*Technol. Rep. Tôhoku Univ.*, 1952, **17** [1], 58-69).—A study is made of the hydrolysis of buffered solutions containing urea, ammonium salts and organic acids in order to utilise the difference of basicity of the rare earths for their separation.

P. N. R. NICHOLS

260. Studies on the separation of the rare earths and their accompanying elements. Part II. Precipitation of rare earths, thorium and zirconium from homogeneous solution. Separation of thorium and zirconium from some lanthanons. Z. Hagiwara (*Technol. Rep. Tôhoku Univ.*, 1952, **17** [1], 70-76).—The relationship between pH and the formation of precipitates of rare earths is studied. The sulphates of the rare earths, thorium and zirconium are added to the buffered solutions described in Part I above, at a temperature of  $90^\circ\text{C}$ . The pH values at which the precipitates are formed are recorded. The results are applied to the separation of thorium and zirconium from some rare earths.

P. N. R. NICHOLS

261. Studies on the separation of the rare earths and their accompanying elements. Part III. Separation of cerium from the rare earths. Z. Hagiwara (*Technol. Rep. Tôhoku Univ.*, 1952, 17 [1], 77-82).—The separation, which is based on the hydrolysis of urea in hot buffered solution, is discussed in detail. The behaviour of the basic precipitate of cerium at various pH values is also studied.

P. N. R. NICHOLS

262. Studies on the separation of the rare earths and their accompanying elements. Part IV. Indirect determination of some elements, such as samarium and yttrium, etc. Z. Hagiwara (*Technol. Rep. Tôhoku Univ.*, 1952, 17 [1], 83-90).—Some lanthanons are precipitated quantitatively by the addition of 8-hydroxyquinoline to a solution buffered with acetic acid and ammonium acetate. On ignition these complexes are converted into the oxides. The determination of samarium and yttrium is made by utilising the difference in weight between the oxide and oxinate.

P. N. R. NICHOLS

263. Methods for analysis of titanium alloys. Colorimetric determination of silicon in titanium alloys. M. Codell, C. Clemency and G. Norwitz (*Anal. Chem.*, 1953, 25 [10], 1432-1434).—In the method given for determining 0.005 to 1.5 per cent. Si in Ti alloys, the Ti is hydrolysed as a crystalline precipitate by heating in a fluoroboric acid solution with  $\text{KMnO}_4$  on a bath of boiling water for 90 min. After filtering the solution,  $(\text{NH}_4)_2\text{MoO}_4$  is added to form the yellow silicomolybdate, tartaric acid is added to prevent interference from moderate amounts of phosphate, and a mixture of 1-amino-2-naphthol-4-sulphonic acid,  $\text{Na}_2\text{SO}_4$  and  $\text{NaHSO}_4$  is added to form a molybdenum blue colour that is measured at 700 m $\mu$ .

O. M. WHITTON

264. Methods for analysis of titanium alloys. Nephelometric and gravimetric determination of small amounts of calcium in titanium alloys. M. Codell, A. Cherney and G. Norwitz (*Anal. Chem.*, 1953, 25 [10], 1434-1436).—In the method described for determining 0.005 to 0.3 per cent. Ca in Ti alloys, the Ti is separated from the Ca by cupferron precipitation and a  $\text{CHCl}_3$  extraction. The Ca is precipitated as  $\text{CaSO}_4$  from a  $\text{CH}_3\text{OH}$  medium, and the Ca is weighed as  $\text{CaSO}_4$  or determined nephelometrically as Ca stearate. Some results are tabulated.

O. M. WHITTON

265. Methods for analysis of titanium alloys. Colorimetric determination of small amounts of aluminium in titanium alloys. M. Codell and G. Norwitz (*Anal. Chem.*, 1953, 25 [10], 1437-1438).—In a method for determining 0.01 to 0.4 per cent. Al in Ti alloys, a cupferron precipitation is made on an 0.02-g aliquot and the solution is filtered. The cupferron in the filtrate is destroyed by fuming with  $\text{HClO}_4$ , the  $\text{HClO}_4$  is driven off, and the Al is determined colorimetrically with aluminon. Some results tabulated indicate possible interferences.

O. M. WHITTON

266. Methods for analysis of titanium alloys. Determination of molybdenum in molybdenum-titanium alloys by precipitation as the sulphide. G. Norwitz and M. Codell (*Anal. Chem.*, 1953, 25 [10], 1438-1441).—A method is described for determining 0.25 to 10 per cent. Mo in Mo-Ti alloys by precipitating it as the sulphide from a  $\text{HF-H}_2\text{SO}_4$  medium. Factors involved in attaining quantitative precipitation of molybdenum sulphide were investigated. Some results are tabulated.

O. M. WHITTON

267. Methods for analysis of titanium alloys. Polarographic determination of molybdenum in titanium alloys. M. Codell, J. J. Mikula, and G. Norwitz (*Anal. Chem.*, 1953, 25 [10], 1441-1443).—A method is described for determining 0.003 to 5 per cent. Mo in Ti alloys. The Mo is extracted with ether from a  $\text{HCl-H}_2\text{SO}_4$  solution; the ether is evaporated off, and the Mo is determined polarographically from a  $\text{HClO}_4$  medium. A study of interfering elements is reported. O. M. WHITTON

268. Methods for analysis of titanium alloys. Determination of phosphorus in titanium alloys. M. Codell and J. J. Mikula (*Anal. Chem.*, 1953, 25 [10], 1444-1446).—In the method given for colorimetrically determining 0.005 to 0.50 per cent. P in Ti alloys, the Mo blue method is used after treating the sample with  $\text{HNO}_3$  and  $\text{HF}$ , evaporating with  $\text{H}_2\text{SO}_4$  to fuming and complexing the Ti with  $\text{NaF}$ . Results are reported and discussed. No interference was observed with Ti samples containing 15% Cr, 10% Mo, 5% Al, 2.5% V, 1% W, 20% Fe, 3%  $\text{SiO}_2$ , and 3% Ni.

O. M. WHITTON

269. Methods for analysis of titanium alloys. Colorimetric determination of small amounts of boron in titanium alloys. M. Codell and G. Norwitz (*Anal. Chem.*, 1953, 25 [10], 1446-1449).—In the method for determining 0.0004 to 0.10 per cent. B in titanium alloys, the sample is dissolved in  $\text{H}_2\text{SO}_4$ , and the Ti and B are oxidised with  $\text{H}_2\text{O}_2$ . The peroxide is destroyed by boiling in the presence of  $\text{FeSO}_4$  as catalyst. The B is distilled as  $\text{B}(\text{OCH}_3)_3$  and then determined colorimetrically with di-anthramide.

O. M. WHITTON

270. The rapid separation and estimation of titanium by means of ethylenediaminetetra-acetic acid. W. F. Pickering (*Anal. Chim. Acta*, 1953, 9 [4], 324-329).—The method of Pribil and Scheider (*Chem. Listy*, 1951, 45, 7) for the estimation of Ti has been modified. An excess (6-7 times the wt. of the cations present) of di-sodium dihydrogen ethylenediaminetetra-acetate is added to an acid solution of the material containing Ti (up to 10 mg).  $\text{NH}_4\text{Cl}$  (1 g) and aq.  $\text{NH}_3$  are added and the vol. is brought to 50 ml; the soln. is cooled, 10 to 20 ml of 10 per cent.  $\text{MgSO}_4$  are added and a cryst. ppt. containing the Ti and Mg is formed ( $\text{Cr}^{III}$  is also pptd. and must therefore be previously oxidised to  $\text{Cr}^{VI}$ ). The ppt. is dissolved from the filter-paper with warm 5 per cent.  $\text{H}_2\text{SO}_4$  and at least 5 ml of 3 per cent.  $\text{H}_2\text{O}_2$  are added through the filter-paper (this tests for completeness of solution). The soln. is made up to a standard vol. and the Ti is estimated colorimetrically (at 425 m $\mu$ ). Fe, Ni, V, Mo, Mn and  $\text{Cr}^{VI}$  do not interfere. The method was also tested with a mixture of rutile-ilmenite sand concentrates and haematite.

E. J. H. BIRCH

271. Potentiometric determination of titanium [together] with vanadium. M. C. Steele and F. M. Hall (*Anal. Chim. Acta*, 1953, 9 [4], 384-388).—V and Ti are determined in steels by removing all the metals except V, Ti and Mo by Hg cathode electrolysis, pptg. Mo with acid sulphide, reducing with Zn and titrating the V and Ti potentiometrically. The steel (2 g) is dissolved in (1 + 1)  $\text{H}_2\text{SO}_4$ , then diluted to 100 ml and aq.  $\text{NH}_3$  is added until the acidity just prevents the formation of a ppt. The solution is electrolysed with a Hg cathode at 8 to 10 volts, 0.3 amp./sq. cm; after completion of electrolysis the soln. is separated from the Hg. The soln. is then reduced with Zn (boiling 30 min. and cooling under  $\text{CO}_2$ ) to give  $\text{Ti}^{III}$  and  $\text{V}^{II}$ . The soln. is

then poured into an excess of ferric alum soln. when  $V^{II}$  is oxidised to  $V^{IV}$ . Potentiometric titration with  $KMnO_4$  gives two steps corresponding to oxidation of  $Fe^{II}$  (equiv. to Ti plus 2/3 V) and  $V^{IV}$  (1/3 V). Alternatively, titration with  $KMnO_4$  to a visual end-point after  $SO_2$  reduction gives the  $V^{IV}$  to  $V^V$  step and after reduction with Zn gives the total Ti plus V. E. J. H. BIRCH

**272. Polarographic reduction of germanium.** A. K. Das Gupta and C. K. N. Nair (*Anal. Chim. Acta*, 1953, **9** [3], 287-291).— $Ge^{IV}$  provides satisfactory polarographic waves in alkaline or feebly acid solutions at 30°C. A solution 0.1 M in respect of  $NH_4Cl$  and 0.5 M in respect of  $NH_3$  is recommended, and 0.01 per cent. of gelatin is added as a maximum suppressor. In a solution M in  $NH_4Cl$  and 0.5 M in  $NH_3$  two waves are obtained, at -1.45 V and 1.7 V, respectively, with reference to the Hg pool anode. W. C. JOHNSON

**273. Spectrophotometric determination of zirconium [and hafnium].** A. D. Horton (*Anal. Chem.*, 1953, **25** [9], 1331-1333).—Zr and Hf are determined spectrophotometrically at 555  $m\mu$  as their 2-(2-hydroxy-3:6-disulpho-1-naphthylazo)benzenearsonic acid complexes.

Standards are prepared as follows: pure Zr metal is dissolved in aq. HCl - NaF, the solution is evaporated and fumed with  $HClO_4$  -  $HNO_3$ ;  $ZrO_2$  is dissolved by fusion in  $Na_2B_2O_7$ ; hafnium mandelate is fumed with  $HClO_4$  -  $HNO_3$ . All solutions are made up to a standard volume with dil. HCl, and suitable diluted aliquots are taken for calibration purposes and for comparison with unknown Zr - Hf samples dissolved in dil. HCl, all solutions having been treated alike with 2-(2-hydroxy-3:6-disulpho-1-naphthylazo)benzenearsonic acid reagent. The colour develops over 2 hr. at room temp. or 5 min. at 75° to 80°C and is stable for 6 hr. Precision is claimed as 5-7 per cent. for 10  $\mu g$  of Zr and 2-9 for 100  $\mu g$ .  $Fe^{III}$ ,  $Cr^{III}$ ,  $Mo^{VI}$ ,  $Ti^{IV}$ ,  $Sr^{II}$ ,  $U^{IV}$ ,  $Cu^{II}$ ,  $ClO_4^-$  interfere only in large excess, but  $Sn^{II}$ ,  $Cr^{VI}$ ,  $PO_4^{III}$  and  $F^-$  interfere at low concentrations. Spectrophotometrically, 2.1  $\mu g$  of Hf  $\equiv$  1  $\mu g$  of Zr. D. A. PANTONY

**274. Mass spectrometric analysis of mixtures containing nitrogen dioxide.** R. A. Friedel, A. G. Sharkey, jun., J. L. Shultz and C. R. Humbert (*Anal. Chem.*, 1953, **25** [9], 1314-1320).—Procedures for determination of  $NO_2$  or  $N_2O_4$  at partial pressures of < 15 and 15-200 microns are described.  $NO$ ,  $N_2O$ ,  $CO_2$ ,  $CO$ ,  $N_2$ ,  $H_2$ ,  $He$ ,  $O_2$  and  $H_2O$  do not interfere. D. A. PANTONY

**275. Polarographic determination of nitrite.** D. Tung-Whei Chow and Rex J. Robinson (*Anal. Chem.*, 1953, **25** [10], 1493-1496).—A method developed for determining nitrite is based on its diffusion enhancement starting at zero applied potential of the polarographic reduction of molybdate. A study of the molybdate-catalysed reduction of nitrite in both sulphate and chloride solutions and of the reduction of nitrate in a chloride medium is described. The current enhancement of both nitrite and nitrate is greater in chloride solutions.

The method is suitable for estimating low concentrations of nitrite and also for determining both nitrite and nitrate in the presence of each other, for which a procedure is proposed. O. M. WHITTON

**276. Colorimetric determination of ammonia and cyanate.** J. M. Kruse and M. G. Mellon (*Anal. Chem.*, 1953, **25** [8], 1188-1192).—The reagent is

prepared shortly before use by mixing 5 parts of a 0.252 per cent. aq. solution of 3-methyl-1-phenyl-5-pyrazolone with 1 part of pyridine containing 0.1 per cent. of bis-(3-methyl-1-phenyl-5-pyrazolone) (prep. described). In the determination of  $NH_3$ , the solution is adjusted to pH 3.7 and buffered with 10 ml of Na acetate - acetic acid buffer, then 0.9 ml of 3 per cent. chloramine-T is added, and after 90 sec., 30 ml of the reagent. After 1 min. the colour is extracted into  $CCl_4$  and the absorbance measured at 450  $m\mu$ . As cyanate is hydrolysed to  $NH_3$  with chloramine-T, the procedure for it is identical, except that  $NH_3$  is first removed by use of suitable ion exchange resins. If cyanide is present, the cyanate is determined by calculation from the absorbances at 450 and 580  $m\mu$ , the ratio for cyanide absorption at the two wavelengths being previously determined. Other interferences are  $Fe$ ,  $Zn$ ,  $Ag$ ,  $Cu$  and thiocyanate. As little as 0.05 p.p.m. of  $NH_3$  can be determined in 1-cm cells. G. P. COOK

**277. Microcrystalloscopic reactions for cyanides.** I. M. Korenman and A. A. Belyakov (*J. Anal. Chem., U.S.S.R.*, 1953, **8**, 168-171).—Cyanides can be detected from the form of their crystalline ppt. with 1:4-ethylbenzylaminophenylmercuriacetate, 1:4-dimethylaminophenylmercuriacetate and 1:2-toluidine-4:6-dimercuriacetate. Certain optical constants are given and photographs of the crystals are reproduced. The min. amount detectable is 0.03  $\mu g$  of  $CN^-$ . To detect  $CN^-$  in solution the third reagent is used and under these conditions the min. amount detectable is 0.25  $\mu g$  of  $CN^-$ . The interference of  $S^{2-}$  is prevented by addition of Pb acetate. Large amounts of  $Cl^-$ ,  $Br^-$ ,  $I^-$ ,  $CNS^-$ ,  $NO_2^-$ , and  $SO_3^{2-}$  do not affect the reaction. G. S. SMITH

**278. New qualitative reaction for thiocyanates.** A. P. Kreshkov, S. S. Vilborg and K. I. Filippova (*J. Anal. Chem., U.S.S.R.*, 1953, **8** [4], 225-227).—Addition of 0.1 N Cu acetate to an equal vol. of a 5 per cent. soln. of aniline in 5 per cent. acetic acid gives a reagent that produces a yellow-brown cryst. ppt. with thiocyanates. According to Korenman ("Microcrystalloscopy," 1947), who proposed the reaction for the detection of Cu, the ppt. is  $[Cu(C_6H_5NH_2)_2 \cdot 5H_2O] \cdot (CNS)_2$ . The test for thiocyanate can be carried out in a test tube, on a spot plate, on filter-paper, or on a microscope slide (sensitivity 1  $\mu g$ ). G. S. SMITH

**279. Volumetric determination of phosphorus content of commercial products [inorganic phosphides].** F. Bermejo Martínez, A. Prieto Bouza, and A. Barrera Ramallo (*Inf. Quim. Anal.*, 1953, **7** [5], 149-153).—To 0.1-0.2 g of the sample in a closed flask is added 20 ml water and then 30 ml 12 N  $H_2SO_4$  is added dropwise. The flask is then heated for 30 min. while the evolved gases are drawn through 25 ml 0.25 N  $Hg(NO_3)_2$ . After filtering, the unchanged  $Hg^{2+}$  is titrated by means of  $KMnO_4$  -  $KCNS$ . The P content of the sample is found by an arbitrary factor; 0.1 equiv.  $Hg(NO_3)_2 \equiv 0.3152 g$  P. D. P. YOUNG

**280. Removal of phosphate ion by ion-exchange.** R. B. Hahn, C. Backer and R. Backer (*Anal. Chim. Acta*, 1953, **9** [3], 223-225).—A column of Amberlite IRA-400 resin 20 cm deep  $\times$  1 cm diam. (30 g) removes up to 300 mg of  $PO_4^{III}$  from a solution of  $NaH_2PO_4$  or  $H_3PO_4$ . By the use of such a column 20-mg quantities, individually or collectively, of the ions of groups III and IV can be separated quant. from 20-100 mg of  $PO_4^{III}$  in a solution 0.25 N in HCl. W. C. JOHNSON

**281. Determination of various phosphates in the presence of one another. I. Modification of the determination of pyrophosphate by the method of Britske and Dragunow.** W. Dewald and H. Schmidt (*Z. anal. Chem.*, 1953, **139** [5], 359-361).—Empirical factors are tabulated to correct for the non-stoichiometric reaction used in the method described previously (*Brit. Abstr. C*, 1951, 94). The factor 13.43 applies over the range 300-700 mg of  $\text{Na}_2\text{P}_2\text{O}_7$ , the sample wt. being taken within this range.

R. E. STANTON

**282. Determination of phosphate by differential spectrophotometry.** A. Gee and V. R. Deitz (*Anal. Chem.*, 1953, **25** [9], 1320-1324).—The method is based upon the yellow complex formed by  $\text{PO}_4^{3-}$  with molybdate and vanadate in acid solution. The complex formation in unknown solutions of  $\text{PO}_4^{3-}$  is adjusted to give an absorbance of  $\approx 2$  at 390 m $\mu$ . The absorbances of these solutions are determined against an accurately known standard  $\text{PO}_4^{3-}$  solution of nearly the same absorbance. The procedure is capable of comparing  $\text{PO}_4^{3-}$  concn. with a precision of 1 part in 3000. Substances (several of which are commonly found in phosphate rocks) that may cause serious interference include  $\text{Fe}(\text{NO}_3)_3$ ,  $\text{Na}_2\text{HAsO}_4$ ,  $\text{H}_3\text{PO}_3$ ,  $\text{K}_2\text{Cr}_2\text{O}_7$ , and  $\text{Na}_2\text{SiO}_3$ .

J. M. JACOBS

**283. Paper chromatographic separation of poly- and meta-phosphates. I. Development of method. II. Quantitative application.** J. P. Ebel (*Bull. Soc. Chim. France*, 1953, **20** [10], 991-998; 998-1000).—An excellent qual. and reasonably accurate quant. ascending chromatographic procedure is developed for the separation of linear polyphosphates (ortho-, pyro-, tri-phosphates and Graham's salt) and cyclic metaphosphates [e.g.,  $(\text{NaPO}_3)_n$ ,  $(\text{NaPO}_2)_n$ ] in mixtures thereof. The structure of each type of phosphate determines its position in the chromatogram; linear and cyclic compounds yield two distinctly separate groups of spots, the migration of the compounds in each group being inversely proportional to mol. wt., although in acid and alkaline solvents the positions of the spots are reversed. Even small differences in composition, e.g.,  $\text{H}_3\text{P}_3\text{O}_9$  and  $\text{H}_3\text{P}_3\text{O}_{10}$ , are reflected by a wide spacing of the corresponding spots. By means of unidimensional chromatography a perfect separation of  $\text{Na}_2\text{HPO}_4$ ,  $\text{Na}_4\text{P}_2\text{O}_7$ ,  $\text{Na}_2\text{P}_2\text{O}_7$ ,  $\text{Na}_2\text{P}_2\text{O}_7$ ,  $\text{Na}_2\text{P}_2\text{O}_7$ , and Graham's salt is attained in an acid solvent (75 ml isopropyl alcohol, 25 ml water, 0.3 ml aq.  $\text{NH}_3$ , 5 g trichloroacetic acid), whilst an alkaline solvent (40 ml isopropyl alcohol, 20 ml isobutyl alcohol, 40 ml water, 1 ml aq.  $\text{NH}_3$ ) will effectively separate  $\text{Na}_2\text{HPO}_4$ ,  $\text{Na}_2\text{P}_2\text{O}_7$ , and Graham's salt only. All six phosphates are effectively separated by two-dimensional chromatography first by the above alkaline solvent and then (after drying the paper) by the acid solvent. The concn. of the phosphate solutions should be  $\approx 5 \mu\text{g}$  of P per 0.02 ml. The paper is sprayed with a solution containing 5 ml  $\text{HClO}_4$ , 10 ml  $\text{N HCl}$ , 25 ml 4 per cent. aq.  $\text{NH}_3$ , molybdate and 60 ml water, dried for 10 min. at 85°C, and finally exposed to  $\text{H}_2\text{S}$  to reveal the blue spots. To determine the P contents  $\approx 16$  sq. cm of paper around each spot is cut out and heated for  $\approx 30$  min. with  $\text{HClO}_4$  plus a few drops of  $\text{H}_2\text{O}_2$  or conc.  $\text{HNO}_3$ ; on cooling, the soln. is diluted with water, boiled slowly for 30 min. to complete the hydrolysis of orthophosphate, and the P is determined by the microphotocolorimetric method of Berenblum (*Brit. Abstr. A I*, 1938, 211). Corrections are made for the P in the area beyond

the cut-out spots. Results are accurate to  $\pm 2$  to 3 per cent. for unidimensional separations, but the errors are greater for two-dimensional separations especially for phosphates, e.g.,  $\text{Na}_2\text{HPO}_4$ , having high  $R_F$  values. Full details are given and 6 chromatograms are shown.

W. J. BAKER

**284. The colorimetric determination of vanadium with o-phenanthroline.** G. Jantzsch and F. Zemek (*Z. anal. Chem.*, 1953, **139** [4], 249-255).—V<sup>V</sup> is reduced to V<sup>IV</sup> with  $\text{HCl}$ . Ammoniacal  $\text{FeCl}_3$  is added, and  $\text{Fe}^{II}$  produced during re-oxidation of V is estimated colorimetrically with o-phenanthroline. This is superior to dimethylglyoxime, which was used originally. A similar electrolytic method is described for determination of V in the presence of up to 1000-fold excess of Fe as in steels. Reduction occurs at an Hg cathode and the  $\text{Fe}^{II}$ -o-phenanthroline complex is formed as before. C. G. TAYLOR

**285. Determination of traces of arsenic in biological materials.** K. Linhardt and I. Lauterbach (*Hoppe-Seyl. Z.*, 1953, **292**, 207-212).—The effect of slight variations in Seifert's procedure for the micro-determination of As was investigated. Provided all the As is reduced and all the resultant  $\text{AsH}_3$  is absorbed by the filter-paper soaked in  $\text{AgNO}_3$  solution, the method is capable of determining 0.05-4.0  $\mu\text{g}$  As.

L. ROWE

**286. Photometric determination of columbium, tungsten, and tantalum in stainless steels.** L. Ikenberry, J. L. Martin, and W. J. Boyer (*Anal. Chem.*, 1953, **25** [9], 1340-1344).—The mixed oxides (obtained by double hydrolysis) are fused with  $\text{NaHSO}_4$  and the melt is leached with 4 per cent. aq.  $\text{NH}_3$  oxalate. To determine the Ta, 10 ml of dil.  $\text{H}_3\text{PO}_4$  (1 + 3) and 20 ml of a solution of 50 g of pyrogallol in 100 ml of water are added to a 50-ml aliquot, the mixture is diluted to 100 ml with 4 per cent. aq.  $\text{NH}_3$  oxalate, the temp. of the soln. is adjusted to  $30 \pm 1^\circ\text{C}$  and, after setting aside for 10 min., the absorbancy is measured with Kromatrol filter 2 (max. transmittance at 430 m $\mu$ ). To determine Nb and W, a 5-ml aliquot of the  $\text{NH}_3$  oxalate leach is evaporated to the fuming point with 10 ml of  $\text{H}_2\text{SO}_4$ , 1 ml of dil.  $\text{H}_3\text{PO}_4$  (1 + 3), and 10 ml of  $\text{HNO}_3$ , then 1 drop of a soln. of 30 g of  $\text{SnCl}_2$  in 100 ml of dil.  $\text{HCl}$  (1 + 4) is added and the mixture is diluted to 100 ml with a solution of 6 g of hydroquinone in 100 ml of  $\text{H}_2\text{SO}_4$ . The absorbancy is measured with Kromatrol photometer filters 3 and 5. From a statistical study of the results of analyses of stainless steel, the reproducibility of the method is  $\pm 0.03$  per cent. for Nb and for W and  $\pm 0.02$  per cent. for Ta. J. M. JACOBS

**287. Sulphur in ores, concentrates, and other metallurgical samples. Routine determination by the combustion method.** W. G. Rice-Jones (*Anal. Chem.*, 1953, **25** [9], 1383-1385).—A rapid combustion method for sulphur determination is described. A 20 to 100-mg 200-mesh sample is heated in a specially designed tube to 2600°F in O and the  $\text{SO}_2$  formed is passed into 0.2 N  $\text{HCl}$  containing starch-KI solution. Sufficient standard  $\text{KIO}_3$  is added to maintain an excess of I during the entry of  $\text{SO}_2$ , and the final excess is titrated with standard  $\text{Na}_2\text{S}_2\text{O}_3$ . A precision of  $\pm 0.2$  per cent. is claimed, and a wide selection of S concentrations in several materials, including those containing Ba, have been tested. Application to routine determinations is discussed in terms of cost and speed. D. A. PANTONY

**288. Colorimetric determination of molybdenum with mercaptoacetic acid.** F. Will III and J. H. Yoe (*Anal. Chem.*, 1953, **25** [9], 1363-1366).—Mo is determined absorptiometrically at 365 m $\mu$  as the thioglycollic acid complex. The  $\text{MoO}_4^{2-}$  (4-15 p.p.m. of Mo) solution, buffered to pH 3.5-4.5, is treated with thioglycollic acid ammonium salt, and the absorbancy is compared with that of standard solutions. Interferences and tolerance limits of several ions are discussed;  $\text{Fe}^{III}$  does not interfere under the experimental conditions and can be determined colorimetrically after adjustment of pH to 8 to 11.

D. A. PANTONY

**289. Indirect complexometric determination of tungsten in minerals.** A. de Sousa (*Anal. Chim. Acta*, 1953, **9** [4], 309-313).—The finely powdered mineral (1 g) (wolframite and scheelite were used in testing the method) is fused with  $\text{K}_2\text{CO}_3$ - $\text{Na}_2\text{CO}_3$  mixture; the W is taken up in water as tungstate and then pptd. as Ca tungstate at pH 8-9. The ppt. is treated with aq. HCl and filtered from the pptd. tungstic acid to give a solution of  $\text{CaCl}_2$ , which after adjustment of pH to 12-13, is titrated with disodium dihydrogen ethylenediaminetetra-acetate with the help of murexide as indicator. The accuracy is better than 0.5 per cent.

E. J. H. BIRCH

**290. Small amounts of uranium in the presence of iron. Colorimetric determination with 8-quinolinol.** L. Silverman, L. Moudy and D. W. Hawley (*Anal. Chem.*, 1953, **25** [9], 1369-1373).—The U solution (10-50  $\mu\text{g}$  of U at pH 8.7-8.9), after treatment by one of several discussed methods for removal of interfering elements and with the Fe converted to ferrocyanide, is treated with 8-hydroxyquinoline.  $\text{CHCl}_3$  is used to extract quantitatively uranium 8-hydroxyquinolate, and the U is determined spectrophotometrically by comparison with standards at 400, 420 or 440 m $\mu$ . Cu, Ni and Co do not interfere unless in high concentration.

D. A. PANTONY

**291. The determination of microgram quantities of fluoride. II. The determination of fluoride in waters and sewage effluent using the aluminium-haematoxylin complex.** J. S. Beveridge, G. J. Hunter and B. J. MacNulty (*Anal. Chim. Acta*, 1953, **9** [4], 330-337).—The Al-haematoxylin method for the estimation of  $\text{F}^-$  (Hunter *et al.*, *Brit. Abstr. C*, 1953, 499) is applied to sea waters, sewage effluent and potable waters. For sea-waters calibration must be carried out in an artificial sea water (composition given) whose  $\text{F}^-$  content is previously determined after separation as  $\text{H}_2\text{SiF}_6$ . Determination in river waters can be carried out directly if  $\text{F}^-$  content is  $>0.1$  p.p.m. The mean  $\text{F}^-$  content for the Atlantic off the British Isles is 1.33 p.p.m.

E. J. H. BIRCH

**292. Quantitative separation of chlorine, bromine, and iodine.** E. Kahane and M. Kahane (*Compt. Rend.*, 1953, **237**, 1244-1246).—The method of separation is based on that recently studied qualitatively by Martin (*Brit. Abstr. C*, 1951, 398), viz., oxidation of HI and HBr by conc.  $\text{HNO}_3$  in the cold by use of a series of wash-bottles through which air or H is passed. Cl $_2$  evolution of which in small amounts is unavoidable, is absorbed in 6.6 N  $\text{HNO}_3$ . The bubblers contain successively: (i) 4 ml of halide solution with an addition of 1 ml of 30 per cent.  $\text{NaNO}_2$  and (to accelerate the oxidation) 5 ml of  $\text{HNO}_3$  ( $d = 1.38$ ), (ii) and (iii) 6.6 N  $\text{HNO}_3$ , (iv) 10 per cent.  $\text{Na}_2\text{SO}_3$  to absorb Br, and

(v) 10 per cent. NaOH to absorb I. Additional  $\text{HNO}_3$  wash-bottles are inserted if the halide concn. is high (2 to 4 N). The separated halogens are determined by standard methods; the results are accurate to  $\pm 1$  per cent.

W. J. BAKER

**293. The micro-estimation of cobalt with triphenylmethylarsonium thiocyanate.** K. W. Ellis and N. A. Gibson (*Anal. Chim. Acta*, 1953, **9** [3], 275-280).—To 10 ml of a solution containing 10 to 100  $\mu\text{g}$  of  $\text{Co}^{++}$  per ml and having a pH of 1 to 4, are added 1 ml of 2 per cent. methyltriphenylarsonium chloride soln. and 1 ml of 25 per cent. aq.  $\text{NH}_4\text{SCN}$ , and the arsonium thiocyanato-cobaltate is extracted with 5-ml and 1-ml portions of *o*-dichlorobenzene. One ml of the reagent soln. is added and the aq. layer is extracted twice more with 1-ml portions of *o*-dichlorobenzene; this treatment is repeated. The combined extracts are diluted to 25 ml and the extinction is determined on a Spekker absorptiometer with a 1-cm cell, H 503 heat filter and 607 orange-red filter. Absorption max. is at 627 (and 590) m $\mu$ . The accuracy is  $\pm 2$  per cent. Ni and Mn interfere only when present in relatively high concn. Interference by  $\text{Fe}^{III}$  is eliminated by adding NaF and that of  $\text{Cu}^{II}$  by adding  $\text{Na}_2\text{S}_2\text{O}_8$ .  $\text{Cr}^{III}$  must be reduced by separation to  $<10$  times the Co concn.  $\text{Mo}^V$  must be oxidised or reduced.

W. C. JOHNSON

**294. Coloration of melted polyphosphates by cobalt, nickel and copper, and an explanation of the reactions in the microcosmic salt bead.** M. Kohn (*Anal. Chim. Acta*, 1953, **9** [3], 226-228).—The colour of melted Na metaphosphate that contains Co, Ni or Cu becomes darker if a small quantity of  $\text{Na}_2\text{CO}_3$  is added in the prep. of the melt. The change of colour is attributed to the formation of polyphosphates, as follows.  $\text{Na}_2\text{CO}_3 + 2\text{NaPO}_3 \rightarrow \text{Na}_4\text{P}_2\text{O}_7 + \text{CO}_2$ .  $\text{Na}_4\text{P}_2\text{O}_7 + 2\text{NaPO}_3 \rightarrow \text{Na}_6\text{P}_3\text{O}_{10}$ . The darker colours are produced for the same reason if oxides of the metals are used instead of salts. The postulated mechanism explains parallel observations with microcosmic salt beads.

W. C. JOHNSON

**295. Heterometric microtitration of nickel with dimethylglyoxime.** M. Bobtelsky and Y. Welwart (*Anal. Chim. Acta*, 1953, **9** [3], 281-286).—The heterometric method (*Bull. Soc. Chim. France*, 1953, **20**, 276, 382 and 687) is applied to the micro-titration of Ni ( $\approx 0.5$  mg) with a solution of dimethylglyoxime, and to the micro-titration of dimethylglyoxime with a solution of  $\text{NiSO}_4$ . (See also *Anal. Abstr.*, 1954, **1**, 296.)

W. C. JOHNSON

**296. Direct heterometric microtitration of nickel with dimethylglyoxime in the presence of other metals.** M. Bobtelsky and Y. Welwart (*Anal. Chim. Acta*, 1953, **9** [4], 374-383).—Solutions of metals of which  $\approx 2$  per cent. is Ni are titrated with 0.01 M dimethylglyoxime from a micro-burette with continuous determination of the optical density with the aid of a red filter. At the end-point the optical density becomes constant at a max. value. Citrate is used as a complexing reagent to prevent interference by Al,  $\text{Fe}^{III}$ , Pb, Cd, Co (if  $<85$  per cent.), Zn, Mg; tartrate prevents interference by Al,  $\text{Fe}^{III}$ , Pb, Cd, Co (if  $<85$  per cent.), Mn, Zn; Na acetate prevents interference by Al, Cr, Cd, Mn, Zn, Mg; and  $\text{Na}_3\text{P}_3\text{O}_{10}$  prevents interference by Al,  $\text{Fe}^{III}$ , Cd, Cu (if  $<87.5$  per cent.), Co (if  $<85$  per cent.), Mn, Zn, Mg. In the presence of citrate and Mn an end-point at half the expected value for Ni is obtained. A table is given of detailed

results with various mixtures of salts. (See also *Anal. Abstr.*, 1954, 1, 295.) E. J. H. BIRCH

297. **Absorptiometric determination of nickel with  $\beta$ -mercaptopropionic acid.** J. B. Lear and M. G. Mellon (*Anal. Chem.*, 1953, 25 [9], 1411-1412).—To 50 ml of solution that has been treated to remove interfering ions, and which contains  $>0.5$  mg of Ni, 10 ml of 1 per cent. aq.  $\beta$ -mercaptopropionic acid are added, the soln. is diluted to  $\approx 90$  ml, the pH is adjusted to  $9.1 \pm 0.5$ , and the vol. is made up to 100 ml. The absorbance is measured at 330 or 410  $m\mu$  against water as a blank. Acetate,  $\text{NH}_4^+$ ,  $\text{Ba}^{++}$ ,  $\text{Ca}^{++}$ ,  $\text{Cl}^-$ ,  $\text{F}^-$ ,  $\text{Mg}^{++}$ ,  $\text{NO}_3^-$ ,  $\text{NO}_2^-$ ,  $\text{PO}_4^{---}$ ,  $\text{K}^+$ ,  $\text{Na}^+$ , and tartrate do not interfere at concn. up to 500 p.p.m. Cu, Fe, Pb, and Mn form ppt. of hydrated oxides when the pH is adjusted to 9, but interference by these metals is avoided by maintaining the Cu at  $< 100$  p.p.m. and Pb and Mn at  $< 50$  p.p.m. As little as 3 p.p.m. of Fe is pptd., but the difficulty is overcome by pptg. the Fe with cupferron and extracting the complex with  $\text{CHCl}_3$ ; only negligible amounts of Ni are lost in the extraction. J. M. JACOBS

298. **A critical evaluation of colorimetric methods for the determination of platinum metals.** F. E. Beamish and W. A. E. McBryde (*Anal. Chim. Acta*, 1953, 9 [4], 349-367).—A review of colorimetric methods for the determination of Pt, Pd, Ir, Rh, Os and Ru. E. J. H. BIRCH

299. **Use of bomb calorimeter with carbonaceous clays.** R. E. King (*Chem. & Ind.*, 1953, [41], 1074).—More consistent data for the calorific values of carbonaceous clays are obtained by ignition (in O under pressure) of  $\approx \frac{1}{8}$ -in. layer of clay in a Pt dish inside the bomb calorimeter, ignition being effected by burning an Fe wire embedded in the clay. Use of a standard fuel is generally unnecessary. Results for Oxford clay are listed. W. J. BAKER

See also Abstracts 220, 318, 320, 328, 354, 376, 377, 396, 421.

### 3.—ORGANIC ANALYSIS

300. **Direct determination of oxygen in organic compounds. Report of the Sub-committee on Analysis of Oxygenated Compounds, Committee on Analytical Research, Division of Refining, American Petroleum Institute.** W. H. Jones (*Anal. Chem.*, 1953, 25 [10], 1449-1452).—Four modifications of the original Unterzaucher method are described for determining 0.01 to 1.0 per cent. O in organic compounds with reasonable accuracy. They satisfactorily obviate the hydrogen effect on the  $\text{I}_2\text{O}_5$ . In the first method, the CO was passed over copper oxide at  $300^\circ\text{C}$ , and the  $\text{CO}_2$  formed was measured manometrically after separation of impurities in cold traps. In the second, the  $\text{I}_2\text{O}_5$  oxidation reaction was used but the  $\text{CO}_2$  formed was measured by absorption in 0.05 N NaOH, precipitation of the carbonate with  $\text{BaCl}_2$ , and back titration of the excess of alkali with 0.025 N HCl by means of a colour indicator. In the third, the liberated I was absorbed in crystals of  $\text{Na}_2\text{S}_2\text{O}_8 \cdot 5\text{H}_2\text{O}$ , and the  $\text{CO}_2$  was determined gravimetrically. In the fourth, the pyrolytic H was removed by diffusion through metallic Pd heated to  $350^\circ\text{C}$  before reaction of the CO with  $\text{I}_2\text{O}_5$ ; the liberated I was collected and titrated with thiosulphate as in the conventional Unterzaucher method. O. M. WHITTON

301. **The origin of some methods of quantitative organic analysis via functional groups.** M. Kohn (*Anal. Chim. Acta*, 1953, 9, [3], 249-255).—Notes

on certain Austrian chemists and their pioneer work in organic analysis: Zeisel (alkoxy determination), Herzog and Hans Meyer (adaptation of Zeisel's method to alkylamino groups) and Huebel (iodine numbers). W. C. JOHNSON

302. **Automatic infra-red punched-card identification of mixtures. Machine method combining use of band wave lengths and intervals of no band.** A. W. Baker, Norman Wright and A. Opler (*Anal. Chem.*, 1953, 25 [10], 1457-1460).—A punched-card system described for analysing mixtures uses the position of bands and regions of no bands in the infra-red spectrum. In an automatic technique involving Hollerith-type cards and the IBM Company's collator, several components of a mixture can be identified with a single pass of cards. Examples describe the identification of mixtures of 20 per cent. *tert*-butyl methyl ketone, 40 per cent. methanol and 40 per cent. acetone; 50 per cent. chloronaphthalene, 25 per cent. 1:3:5-trichlorobenzene and 25 per cent. *o*-chloroethylbenzene; 20 per cent. di-(*p*-chlorophenyl) sulphone, 40 per cent. nitroethane and 40 per cent. 1:2-dimethoxyethane; and 25 per cent. methylene bromide, 25 per cent. methylene chlorobromide and 50 per cent. methylene iodide. O. M. WHITTON

303. **Analysis of organic hydroperoxides in the presence of hydrogen peroxide.** H. Bruschweiler, G. J. Minkoff and K. C. Salooja (*Nature*, 1953, 172, 909).—The identification and estimation of organic hydroperoxides in the presence of hydrogen peroxide by two different techniques is described. The first, mainly qual., is by paper chromatography. The second, which is quant., is polarographic. In alkaline medium (0.1 M  $\text{Li}_2\text{SO}_4$ , 0.01 M LiOH), hydrogen peroxide gives two waves, one anodic ( $E_1 = -0.1$  V) and one cathodic ( $E_1 = -0.9$  V). The diffusion currents of hydrogen peroxide corresponding to these steps have equal and opposite values. Organic hydroperoxides contribute to the second step only, and with different diffusion currents. When the concn. of hydrogen peroxide is much greater than that of the organic peroxides, the hydrogen peroxide is precipitated in an alkaline medium with 0.01 M lanthanum acetate, when an irreducible basic peroxide of lanthanum is formed. The lower limit of detection is  $\approx 10^{-3}$ - $10^{-6}$  M peroxide. I. JONES

304. **Saturated hydrocarbons of high molecular weight.** S. A. Francis (*Anal. Chem.*, 1953, 25 [10], 1466-1470).—A method is developed for the structural group analysis of saturated hydrocarbons of high molecular weight. On the basis of infra-red absorption data for 28 pure saturated hydrocarbons, it is concluded that methyl groups can be determined from the 7.254- $\mu$  absorptivity, paraffinic methylene groups from the 3.382 and 3.418- $\mu$  absorptivity, and cyclopentyl and cyclohexyl methylene groups from the 3.382 and 3.418- $\mu$  absorptivities after correcting for the absorption due to methyl and paraffinic methylene groups at these two spectral positions. O. M. WHITTON

305. **Infra-red band correlations for some substituted olefines.** R. E. Kitson (*Anal. Chem.*, 1953, 25 [10], 1470-1472).—The infra-red spectra of more than 100 substituted olefines, principally halo- and cyano-olefines containing from 3 to 5 C atoms have been recorded. The effects of such substituents on the out-of-plane bending vibrations and the presence of other bands which may be characteristic of these compounds are presented.

O. M. WHITTON

## 306. Methods of analysis of halogenated ethers.

**I. Analysis of  $\alpha$ -halogen-dialkyl and ethylaryl ethers.** M. F. Shostakovskiy and A. V. Bogdanova (*J. Anal. Chem., U.S.S.R.*, 1953, 8 [4], 231-234).—Compounds of the type  $\text{RCH}_2\text{CHXOR'}$  where  $\text{R} = \text{H}$  or  $\text{CH}_3$ ,  $\text{X} = \text{Cl}$  or  $\text{Br}$ , and  $\text{R'} = \text{Alkyl}$  or  $\text{Aryl}$  are hydrolysed instantaneously at room temp. to  $\text{RCH}_2\text{CHO}$ ,  $\text{HX}$  and  $\text{R'OH}$  by excess of water. Detection of the compounds can be based on tests for aldehyde, halide, or acid. They can be determined by hydrolysis followed by titration with 0.1 N NaOH, titration with 0.1 N  $\text{AgNO}_3$ , or titration of the aldehyde with bisulphite. A number of different compounds were determined in this way with excellent results. G. S. SMITH

**307. Identification of glycol and glycol derivatives.** H. Böhm and H. Opfer (*Z. anal. Chem.*, 1953, 139 [4], 255-263).—Pseudosaccharin chloride derivatives, of simple glycols, glycol ethers, and glycol esters are prepared from  $\text{CHCl}_3$  or  $\text{CHCl}_3\text{-C}_6\text{H}_5\text{N}$  solution. The compounds are characterised by m.p., elementary analysis, and eutectic temp. with Salophen, dicyandiamide, benzil, acetanilide, azobenzene, or phenacetin. The prep. of pseudosaccharin chloride and two general methods for preparing these derivatives are described. C. G. TAYLOR

**308. Paper chromatography in analysis of food [and commodities]. II. Detection and testing for purity of glycols and related compounds.** K. G. Bergner and H. Sperlich (*Z. Lebensmitt. Untersuch. u. Forsch.*, 1953, 97, 253-263).—Polyhydroxy alcohols, including glycols, can be separated from reducing sugars and impurities, and from one another by use of  $\text{CHCl}_3$ -ethanol (4 + 1) as ascending solvent, and detected by spraying with a (9 + 1) mixture of 5 per cent. aq.  $\text{AgNO}_3$  and 25 per cent. aq.  $\text{NH}_3$ . The required min. concn. for this reaction, viz., 5 per cent. for glycerol and butane-1:2:4-triol, 10 per cent. for 1:2-glycols, and 20 per cent. for 1:3- and 1:4-diols, may be attained, if necessary, by extraction with ether. Butylene-glycols and propylene-glycols can be separated from the other above-mentioned compounds, and from one another, by multiple development by means of water-saturated ether as solvent. Chromatograms can be fixed by treatment with 10 per cent. thiourea in 0.1 N  $\text{H}_2\text{SO}_4$ . Glycol esters are identified by chromatography after partial hydrolysis with 4 N HCl at 200°C (in capillary tubes). Directions are given for dealing with commercial glycols, beverages, and toothpastes (see also *Dtsch. Lebensmitt. Rdsch.*, 1951, 47, 134).

P. S. ARUP

**309. Infra-red spectra of sugar anomers.** R. L. Whistler and L. R. House (*Anal. Chem.*, 1953, 25 [10], 1463-1465).—Examination of the infra-red absorptions of monosaccharides (L-arabinose, D-xylose, D-lyxose, D-glucose, D-mannose, D-galactose, D-fructose, L-rhamnose, L-fucose) and their derivatives suggests that certain regions of absorption are characteristic of the configuration of the anomeric carbon and may be used to identify its  $\alpha$ - or  $\beta$ -structure. As the absorptions for different sugars differ in magnitude and in spectral position, the sugar type and reference absorptions must be known in order to assign the anomeric configuration. The method of producing the spectra is described and the results are tabulated. O. M. WHITTON

**310. Paper electrophoresis of sugars at high potential gradients.** D. Gross (*Nature*, 1953, 172,

908-909).—The separation of sugars by electrophoresis on paper with potential gradients of 40-60 V per cm by use of polythene and copper plates for the dissipation of the generated heat is described. Both raffinose and stachyose travel faster towards the anode than sucrose; this is contrary to their behaviour on a paper chromatogram. Glucose and fructose are not separated under the conditions described, but can be resolved at a lower pH. Kestose, a trisaccharide, can be easily separated from raffinose, contrary to chromatographic behaviour. Its separation from sucrose is sufficiently clear provided their proportions are not too unfavourable. I. JONES

**311. A colorimetric estimation of sugars using benzidine.** J. K. N. Jones and J. B. Pridham (*Nature*, 1953, 172, 161).—A quantitative technique for estimating sugars and their methylated derivatives (except ketoses) by means of benzidine in acetic acid is given. It appears that mol. equivalents of benzidine and sugar (having a free or easily accessible aldehyde group) combine to give a coloured compound. The advantages of the method are in the mild nature of the reagents and in the wide range of sugars that can be estimated.

E. M. RATTENBURY

**312. Qualitative and quantitative colorimetric determination of heptoses.** Z. Dische (*J. Biol. Chem.*, 1953, 204 [2], 983-997).—The reactions of various heptoses with (i) orcinol- $\text{FeCl}_3$  (Bial's reaction; 3 modifications), (ii)  $\text{H}_2\text{SO}_4$ , (iii) cysteine- $\text{H}_2\text{SO}_4$ , (iv) diphenylamine-HCl, (v) carbazole- $\text{H}_2\text{SO}_4$ , and (vi) phloroglucinol are investigated, and the absorption spectra of the coloured products measured. Reaction (i) can be used to distinguish aldo- and keto-heptoses. Only (i), (iii) and (iv) show promise for quant. analysis of heptoses in the presence of other sugars, and accurate determination is possible only when a single known heptose is being analysed. Prior hydrolysis of heptose esters may be necessary, and allowance must be made for the colours produced by the action of  $\text{H}_2\text{SO}_4$  on other organic materials present during analysis by the running of suitable blanks. C. E. SEARLE

**313. Colorimetric estimation of formaldehyde by means of Hantzsch reaction.** T. Nash (*Biochem. J.*, 1953, 55 [3], 416-421).—A colour reaction for formaldehyde is described which depends on the synthesis of diacetyldihydrolutidine from acetylacetone and formaldehyde in the presence of an excess of ammonium salt, when a yellow colour with mol. extinction of 8000 at 412 m $\mu$  is formed. The method is suitable for concn. of  $10^{-4}$  M of formaldehyde. The conditions are mild enough to allow its use with living material, and its sensitivity and specificity are comparable with those of other reactions requiring more severe conditions.

B. VINEY

**314. Separation and estimation of organic acids on paper chromatograms.** F. A. Isherwood and C. S. Hanes (*Biochem. J.*, 1953, 55 [5], 824-830).—Qualitative examination of a number of common aliphatic acids on a paper chromatogram with *n*-propanol-conc. aq.  $\text{NH}_3$  as solvent, shows that many of them can be readily separated.  $R_F$  values are given and the results are also expressed in terms of  $\log_{10} \left( \frac{1}{R_F} - 1 \right)$  plotted against the number of C atoms in the molecule, to throw light on relationships in the chromatographic behaviour of the acids. A quantitative method is described

for the estimation of the ammonium salts of the acids after separation on a similar paper chromatogram. It is based on the effect of the ammonium salts on a thymol blue-glycine reagent that is insensitive to free  $\text{NH}_3$ . The change in colour of the thymol blue is inversely proportional to the concn. of the salt from about 80 to about 20 per cent. neutralisation of the reagent. The accuracy of the method (single determinations) is about  $\pm 10$  per cent. when  $50 \mu\text{g}$  of organic acid are present.

I. JONES

**315. Paper chromatography of glutamic and aspartic acids.** Reinhard Koch and H. Hanson (*Hoppe-Seyl. Z.*, 1953, **292**, 180-183).—It has been shown that 0.1-0.01 *M* solutions of glutamic and aspartic acids dissolved in a (3 + 1) mixture (by vol.) of ethanol and *N* HCl, as used for chromatographic standards, are esterified upon storage. The extra spots found with such old solutions by both butanol-acetic acid and phenol solvent systems, correspond to the glutamic acid- $\gamma$ -ethyl ester and the aspartic acid- $\beta$ -monoethyl ester.

L. ROWE

**316. Infra-red spectrometric analysis of mixtures of phenol and cresols.** M. Auméras, R. Minangoy, L. Bonnot and B. Laugrost (*Bull. Soc. Chim. France*, 1953, **20** [10], 924-927).—Mixtures of phenol and *o*-, *m*-, and *p*-cresols are analysed quantitatively by determining the optical density of 20 per cent. soln. in  $\text{CS}_2$ , either by the method of base lines (*Brit. Abstr. B II*, 1941, 105) or by means of two cells of different thicknesses (*Brit. Abstr. A I*, 1937, 344). A mirror-spectrometer with NaCl prism and Wadsworth attachment is used. The percentage of *o*-cresol in a mixture of the three isomers is calculated from the optical density-concn. curve. For the analysis of a mixture of the four phenols the optical densities are measured at the 4 critical wavelengths 9.3, 12.8, 13.3 and  $12.0 \mu$  and are then used to establish a system of linear equations comprising numerical coeff. obtained by spectrophotometric calibration of the pure components. These equations are resolved mathematically or by a calculating machine so as to give the percentage of each constituent within an accuracy of  $\approx 1$  per cent.

W. J. BAKER

**317. Use of the Lunge nitrometer for determining nitrosamines, nitramines and alkyl nitrates.** B. V. Ioffe and Z. I. Sergeeva (*J. Anal. Chem., U.S.S.R.*, 1953, **8** [4], 235-237).—Aliphatic and alicyclic nitrosamines and nitramines do not react with  $\text{Hg}$  and conc.  $\text{H}_2\text{SO}_4$ . Methyl nitrate can be determined satisfactorily by the nitrometer, but ethyl nitrate gives low results:  $\approx 50$  per cent. of the correct value when alone and  $\approx 80$  per cent. when in 75 per cent. soln. in ethanol. The determination of  $\text{HNO}_3$  in presence of large amounts of organic compounds is of no value unless experimental proof of its applicability in any particular instance is available; thus, results are satisfactory with methanol and dialkyl-nitrosamines but not with ethyl, isopropyl and isobutyl alcohols.

G. S. SMITH

**318. The titration of Complexone III with thorium nitrate at pH 2.8-4.3.** K. Ter Haar and J. Bazen (*Anal. Chim. Acta*, 1953, **9** [3], 235-240).—Complexone III, the di-sodium salt of ethylenediaminetetra-acetic acid, is titrated with 0.05 *M*  $\text{Th}(\text{NO}_3)_4$  in solutions buffered at pH 2.8 to 4.3. In this pH range comparatively few metal ions interfere by forming complexes. At pH 4.3 the titration is carried out at the b.p. Alizarin-S is used as indicator.

W. C. JOHNSON

**319. Qualitative organic micro-analysis based on physical constants. Identification of amino acids.** A. Lacourt, G. Sommereyns, C. Francotte and N. Delande (*Nature*, 1953, **172**, 906-907).—The identification of amino acids by the measurement of two refractive indices, one at right angles to the other, is described. The measurements can be made on a single crystal and the accuracy is  $\pm 0.001$ . A table of *n* for a number of amino acids is given. A table of m.p. of binary eutectic mixtures of amino acids is also given.

I. JONES

**320. 2 : 6-bis(2-Pyridyl)pyridine [2 : 6-Di-(2-pyridyl)pyridine] and alkyl derivatives.** Their properties in the formation of ferrous and cobaltous coloured complex cations. D. H. Wilkins and G. Frederick Smith (*Anal. Chim. Acta*, 1953, **9** [4], 338-348).—The absorption max. with 2 : 6-di-(2-pyridyl)pyridine (terpyridine) of the  $\text{Fe}^{II}$  complex occurs at  $552 \mu$  ( $\epsilon$ , 12,500), and of  $\text{Co}^{II}$  at  $444 \mu$  ( $\epsilon$ , 1420) and  $506 \mu$  ( $\epsilon$ , 1240); with 4-phenyl-2 : 6-di-(2-pyridyl)pyridine (terosine) the max. of  $\text{Fe}^{II}$  is at  $469 \mu$  ( $\epsilon$ , 22,000), and of  $\text{Co}^{II}$  at  $452 \mu$  ( $\epsilon$ , 2630) and  $517 \mu$  ( $\epsilon$ , 2690); and with 2 : 6-di(4-methyl-2-pyridyl)-4-phenylpyridine (terosole) the max. of  $\text{Fe}^{II}$  is at  $575 \mu$  ( $\epsilon$ , 26,300) and of  $\text{Co}^{II}$  at  $457 \mu$  ( $\epsilon$ , 2440) and at  $519 \mu$  ( $\epsilon$ , 2514). All the ligands become progressively more insol. in water as the mol. wt. increases, but their hydrochlorides are sol. None of these ligands forms a complex with  $\text{Cu}^I$ . The complexes are more easily extracted with an immiscible alcohol the higher the mol. wt. Terosole forms an extremely insol. perchlorate even in the presence of  $\text{HCl}$ ,  $\text{HNO}_3$  or  $\text{H}_2\text{SO}_4$ . The ferrous-terosole complex loses  $< 1$  per cent. of its colour intensity after extraction and keeping 130 hr., and the colour withstands boiling at pH 2-5, although it fades at pH 1 in 5 min. The cobaltous-terosole complex is somewhat less stable. Interference with the Fe reaction with terosole (the most sensitive of the reagents) is occasioned by  $\text{Cu}^+$ ,  $\text{Ni}^{2+}$ ,  $\text{Co}^{2+}$ ,  $\text{F}^-$  and  $\text{ClO}_4^-$ , but  $\text{Mn}^{2+}$ ,  $\text{Al}^{3+}$ ,  $\text{Cr}^{3+}$ , alkali and alkaline earth metals,  $\text{PO}_4^{3-}$ ,  $\text{P}_2\text{O}_7^{4-}$ , oxalate,  $\text{Cl}^-$ ,  $\text{NO}_3^-$ ,  $\text{AcO}^-$  and  $\text{SO}_4^{2-}$  do not interfere. The preparation of pure  $\text{CoSO}_4$  is described and a method for determining Fe in presence of Co is described in which the  $\text{Co}^{II}$  is first complexed with ethylenediamine and the colour for the Fe obtained with terosine. The effect of substituents at various points on the 2 : 6-di-(pyridyl)pyridine skeleton is discussed.

E. J. H. BIRCH

**321. Catalytic effect of molybdates on hydrolysis of organic phosphates.** H. Weil-Malherbe (*Biochem. J.*, 1953, **55** [5], 741-745).—When solutions of org. phosphates were heated with molybdate, estimations of inorg. phosphate by direct colorimetry and by colorimetry after previous pptn. did not agree. This is now shown to be observed only when a blue colour develops during exposure to heat. The effect is similar if the solution contains, e.g., either fructose phosphate or a mixture of fructose and phosphate, and it is therefore unlikely to be caused by the formation of organo-molybdophosphate complexes. It is suggested that the effect is due to 3 factors. (i) If magnesia mixture is added to the reduced molybdophosphate complex and the resulting ppt. is analysed colorimetrically, recoveries of inorg. phosphate are low. (ii) Free molybdic acid is reduced, giving Mo blue. Contrary to prevailing opinions, molybdenum-blue is extracted from acid soln. by isobutyl alcohol. (iii) The specific extinction coeff. is about 4 to 5 times higher when

the colour of the reduced molybdophosphate complex is developed at 100° C than it is when developed at room temp. This is true for most reducing agents except  $\text{SnCl}_2$ , with which a higher extinction is obtained at room temp.

B. VINEY

**322. Quantitative fractionation of saturated and unsaturated fatty acids by means of permanganic hydroxylation and invert-phase partition chromatography.** P. Savary and P. Desnuelle (*Bull. Soc. Chim. France*, 1953, **20** [10], 939-945).—Mixtures of  $\approx 50$  mg of saturated fatty acids ( $\text{C}_{10}$ - $\text{C}_{18}$ ) with unsaturated acids, etc., are quant. analysed by invert-phase partition chromatography (solid support, silicone-kieselguhr; fixed phase, cyclohexane or light petroleum; mobile phase, 20 per cent. aq. acetone). Interference caused by simultaneous presence of oleic and palmitic acids, or of linoleic and myristic acids, is controlled by converting the unsaturated acids into hydroxy-acids by means of 0.03 N  $\text{KMnO}_4$ . Di- and tetra-hydroxystearic acids migrate very rapidly along the chromatographic column (13.5 mm diameter) of cyclohexane or light petroleum and are finally fractionated with other columns containing purified castor oil as fixed phase. Separation is quant. satisfactory (within 1 to 3 per cent.) when only 3 or 4 well-defined peaks are involved, but when there is  $<5$  to 10 per cent. of one constituent the error may be 4 to 6 per cent. The method is applicable to natural fats.

W. J. BAKER

**323. Spectrophotometric determination of cholesterol and triterpene alcohols in wool wax.** Francis E. Luddy, Arthur Turner, jun. and John T. Scanlan (*Anal. Chem.*, 1953, **25** [10], 1497-1499).—In the spectrophotometric method described for determining cholesterol and triterpene alcohols in wool wax, 1:4-dioxan is added to the reagent to reduce the rate of colour development and permit operation at room temperature. By determining the triterpenes at the secondary maximum at 550  $m\mu$  and cholesterol at 630  $m\mu$ , a single solution can be used for both. Corrections are made for overlapping absorption bands in both cases. Modifications are described to make the method applicable to the unsaponified wool wax esters, to the mixture of free alcohols obtained by saponifying wool wax, or to precipitated cholesterol digitonide.

O. M. WHITTON

**324. Determination of a hindered phenol in lubricating oils by infra-red absorption. 4-Methyl-2:6-di-tert-butylphenol.** E. Poti, L. L. Gent, R. C. Pomatti and H. Levin (*Anal. Chem.*, 1953, **25** [10], 1461-1463).—An infra-red method for determining 2:6-di-tert-butyl-4-methylphenol in both used and unused industrial lubricating oils is described. The phenol is quantitatively eliminated from the oils by heating, and the difference in baseline absorbancies determined in the 11-63- $\mu$  region between the heated sample and the original sample is multiplied by a factor established experimentally on standard blends. The reproducibility of this test, based on used blends, is characterised by a standard deviation of 0.01 per cent. phenol.

O. M. WHITTON

**325. Properties of high boiling petroleum products. Nonbiological laboratory methods for predicting carcinogenicity.** L. T. Eby, William Priestley, jun., J. Rehner, jun., and Maynard E. Hall (*Anal. Chem.*, 1953, **25** [10], 1500-1507).—Non-biological methods described and discussed for predicting tumour potency of high boiling petroleum products are a u.v. absorption method, a simplified u.v.

absorption method, caffeine number (u.v. absorption of an aq. caffeine soln.) high aromatics characterisation, and polarographic methods. Analytical data are tabulated.

O. M. WHITTON

**326. Radiochemistry in paint research.** D. F. Rushman (*J. Oil Col. Chem. Ass.*, 1953, **36**, 352-372).—Analytical applications of radiochemical methods are reviewed *inter alia*. Original data are quoted to show that an accuracy of 1 per cent. or better may be attained in the determination of K by measuring the  $\beta$ -activity of the naturally present  $^{40}\text{K}$ . The method has been applied to potassium zinc chromate pigments and to Prussian blue. More accurate results are obtained if the K is brought into solution, but direct determination in the dry powders is also possible. Other analytical techniques described are the determination of trace elements (e.g., As in Ge) by pile radioactivation, the analysis of fatty acid and/or triglyceride mixtures by the isotopic dilution method (an example showing an accuracy of 4 per cent. is quoted), the micro-analysis of fatty acids by paper chromatography followed by conversion to the  $^{60}\text{Co}$  salts, and the determination of the composition of the surface of pigment (or other crystalline) particles by radioactive isotope exchange.

D. R. DUNCAN

**327. Observing the end point in tests of latex mechanical stability.** Alfred C. Meyer (*Anal. Chem.*, 1953, **25** [9], 1348-1351).—A new technique, involving only a minor modification in the end-point determination by the A.S.T.M. method of testing the mechanical stability of natural rubber latex, is described. Particle formation during a test is studied throughout the periods of incipient and complete coagulation by means of successive dips with narrow 80-mesh Monel screens, which are then washed free from adhering latex. Variance analysis shows less error than with the currently used rod dipping method on unknown samples.

G. P. COOK

See also Abstracts 223, 228.

#### 4.—BIOCHEMISTRY INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Blood, Bile, Urine, etc.

**328. A micromethod for the determination of serum calcium.** F. W. Fales (*J. Biol. Chem.*, 1953, **204** [2], 577-585).—In determining serum calcium by titration with di-sodium dihydrogen ethylenediaminetetra-acetate and with ammonium purpurate as indicator, the end-point is determined graphically from spectrophotometric readings at 620  $m\mu$  during titration. This gives a much more accurate end-point than does visual observation. The method is used for both micro- and macro-determinations.

C. E. SEARLE

**329. A method for the determination of ergothioneine in blood.** D. B. Melville and R. Lubschetz (*J. Biol. Chem.*, 1953, **200** [1], 275-285).—Ergothioneine is determined colorimetrically by coupling with diazotised sulphanilic acid in the presence of strong alkali. The diazo reaction is carried out on blood filtrates from which interfering substances have been removed by means of ion-exchange resins and org. solvent extraction. The procedure is as follows. One ml of heparinised blood is added to a 12-ml graduated centrifuge tube containing 10 ml

of 0.9 per cent. NaCl soln. The solutions are mixed and centrifuged, and the supernatant diluted plasma is discarded. Water is then added to bring the vol. to 1 ml and to lake the cells. To this lake is added 0.5 ml of a fresh 3.5 per cent. soln. of glutathione, and 25 mg Na dithionite, to prevent loss of ergothioneine during pptn. of the proteins. After mixing the contents of the tube with 5.0 ml of water, the proteins are pptd. by addition of 0.5 ml of 35 per cent. trichloroacetic acid. The mixture is stirred, centrifuged, and the supernatant liquid is extracted with 15 ml  $\text{CHCl}_3$  and shaken for 10 min. with 4 ml of moist Amberlite IRA-410 resin. The resin is removed by filtering through cotton wool. A 3-ml aliquot of the filtrate is added to a calibrated photometer tube, cooled in an ice-bath, and mixed with 0.5 ml of an alkaline buffer solution containing 200 g  $\text{Na}_2\text{CO}_3$  and 57 g Na citrate dihydrate per litre. To this solution is added 0.5 ml of diazotised sulphanilic acid freshly prepared by adding 1 vol. of cold 8 per cent.  $\text{NaNO}_2$  to 10 vol. of cold 0.2 per cent. sulphanilic acid in 1 per cent. HCl. After keeping the mixed solutions in an ice bath for 45 sec., 1.0 ml of 19 N NaOH is added rapidly, the solutions are mixed, and the optical density is measured at 540 m $\mu$ . A blank is prepared from 2.5 per cent. trichloroacetic acid treated in the same manner as the filtrate from the resin. The method is accurate and is sensitive to 10  $\mu\text{g}$  ergothioneine per ml of blood. All mammalian bloods examined give good recoveries of added ergothioneine, and the method is also applicable to certain other tissues. D. V. PARKE

**330. The direct determination of phosphatidyl ethanolamine and phosphatidyl serine in plasma and red blood cells.** J. Axelrod, J. Reichenhalt and B. B. Brodie (*J. Biol. Chem.*, 1953, **204** [2], 903-911).—Phospholipids are extracted from plasma and red blood cells and hydrolysed with alkali. Ethanolamine and serine are converted by dinitrofluorobenzene to their coloured dinitrophenyl deriv., which are separated by means of the solubility of dinitrophenyl ethanolamine in  $\text{CHCl}_3$  and estimated spectrophotometrically at 420 m $\mu$ . The specificity of the method is investigated by means of counter-current distribution.

C. E. SEARLE

**331. Comparison of methods for the estimation of vitamin A in calf plasma.** J. E. Avampato and H. D. Eaton (*J. Dairy Sci.*, 1953, **36**, 783-793).—A comparison has been made of the following methods of determining vitamin A in bovine blood: Kimble's  $\text{SbCl}_5$  method (*J. Lab. Clin. Med.*, 1939, **24**, 1055), Sobel's glycerol dichlorohydrin method (*J. Biol. Chem.*, 1947, **171**, 617), and the spectrophotometric method. The effect of saponification, with and without separation of carotene from vitamin A by pptn. from aq. alcohol solution or by chromatography, has also been determined. S. C. JOLLY

**332. Colorimetric method for the determination of fatty acids in blood by oxidation with dichromate.** A. C. Kibrick and S. J. Skupp (*Arch. Biochem. Biophys.*, 1953, **44** [1], 134-139).—A method is described for the colorimetric determination of fatty acids by oxidation for 20 min. at 15-lb pressure with a solution of  $\text{K}_2\text{Cr}_2\text{O}_7$  in 74 per cent.  $\text{H}_2\text{SO}_4$ . After cooling, the concn. of  $\text{H}_2\text{SO}_4$  is brought to 53 per cent. by dilution with water and the colour of the soln. is compared with a standard at 600 m $\mu$ . The method is applied to the determination of the total fatty acids of blood plasma, and the results

obtained are in good agreement with those found by estimation of the fatty acid esters with iron perchlorate. The advantages of the method are discussed. D. BAILEY

**333. The chromatographic separation of free and combined plasma cholesterol.** Lynda M. H. Kerr and W. S. Bauld (*Biochem. J.*, 1953, **55** [5], 872).—Chromatographic separation of cholesterol from its esters has been achieved on  $\text{Al}_2\text{O}_3$  columns by the use of light petroleum to extract the cholesterol esters and benzene for the free cholesterol. Twelve separate extracts of the same plasma gave a mean value of 153 mg/100 ml (range 143-162 mg/100 ml) for ester cholesterol and a mean value of 51 mg/100 ml (range 45-57 mg/100 ml) for free cholesterol. The accuracy is not as great as that of the Schoenheimer-Sperry method (*J. Biol. Chem.*, 1934, **106**, 745) but is regarded as satisfactory for most clinical purposes.

An acetone-ethanol extract of 0.2 ml plasma was used, and eluted with 30 ml light petroleum followed by 30 ml benzene. It is shown that cholesterol esters may be estimated directly by the Liebermann-Burchard reaction. N. E.

**334. Photo-electric estimation of serum albumin on paper.** M. Wenzel and H. Hanson (*Hoppe-Seyl. Z.*, 1953, **292**, 137-138).—Serum albumin when separated by paper electrophoresis is normally estimated after application of a dye, e.g., bromophenol blue. This photo-electric estimation is shown to be valid as the dye formed obeys the Beer-Lambert law. L. ROWE

**335. Determination of glucose in urine.** D. J. De Jong (*Pharm. Weekbl.*, 1953, **88** [45/46], 776-777).—The author's Cu reagent, 2 ml of which are titrated at the b.p. with the urine sample (*cf. Brit. Abstr. A*, 1932, 295), is modified for use with urine of high glucose content as follows: 8.750 g K tartrate and 3.5 g KOH dissolved in water and made up to 25 ml are poured on to 75 g KCNS; this solution is mixed with a solution of 6.5 g of copper sulphate in water made up to 25 ml and 17.5 g of glycerol, and the mixture is boiled and filtered through sintered glass. Of this reagent, 2 ml = 13.5 mg of glucose. A reagent suitable for use with normal urine (2 ml = 4 mg of glucose) is made by using 45 and 48 ml of water, respectively, in preparing the above solutions. P. S. ARUP

**336. A chromatographic study of uroporphyrins from cases of cutaneous, acute and congenital porphyria.** J. Canivet and C. Rimington (*Biochem. J.*, 1953, **55** [5], 867).—Chromatographic methods have been applied to the study of urinary uroporphyrins from seven cases of cutaneous porphyria. In six cases the predominant isomer was uroporphyrin-I but in one case approx. 75 per cent. uroporphyrin-III was found. Two cases of acute porphyria contained 75 to 80 per cent. uroporphyrin-III and 20 to 25 per cent. uroporphyrin-I, while two cases of congenital porphyria contained about 95 per cent. uroporphyrin-I. N. E.

**337. A method of counting radiosulphur in liquid samples, and its application to the determination of  $^{35}\text{S}$  excretion following injection of  $^{35}\text{SO}_4$ .** M. Walser, A. F. Reid and D. W. Seldin (*Arch. Biochem. Biophys.*, 1953, **45** [1], 91-96).—Total  $^{35}\text{S}$  is measured in liquid samples contained in stainless steel cups roofed by thin aluminium foil and designed for use in a gas-flow counter. An example is included of a study of urinary and faecal excretion

following radiosulphate injection into a normal human subject.

H. H. HODGSON

**338. Ultramicrodetermination of glycogen in liver. Comparison of the anthrone and reducing-sugar methods.** J. Fong, F. L. Schaffer and P. L. Kirk (*Arch. Biochem. Biophys.*, 1953, **45**, 319-326).—The colorimetric determination of glycogen with anthrone reagent and the microgram reducing-sugar method involving  $\text{Fe}(\text{CN})_6^{4-}$  oxidation and  $\text{Ce}(\text{SO}_4)_2$  titration have been modified for use with as little as 1  $\mu\text{g}$  of glycogen (as glucose). The second method shows greater precision. The content of glycogen does not vary greatly from site to site within one lobe of liver.

H. G. REES

**339. A rapid method for the determination of dihydroxyphenylalanine decarboxylase in animal tissues.** L. S. Dietrich (*J. Biol. Chem.*, 1953, **204** [2], 587-591).—Tissue homogenates are incubated for 15 min. at 37°C with DL-dihydroxyphenylalanine (I) in a phosphate buffer, pH 6.9, saturated with octyl alcohol. After heating, cooling and centrifuging, the supernatant liquid is chromatographed on "Permutit" to separate hydroxytyramine from unchanged I, and the former is estimated colorimetrically with Folin's phenol reagent. In the absence of octyl alcohol recoveries of hydroxytyramine are poor owing to monoamine oxidase activity. In mice, the highest I-decarboxylase activities are found in kidney and liver.

C. E. SEARLE

**340. Method for the determination of hexosamines in tissues.** N. F. Boas (*J. Biol. Chem.*, 1953, **204** [2], 553-563).—A number of substances produce interfering colours in the hexosamine determination proposed by Elson and Morgan (*Biochem. J.*, 1933, **27**, 1824). These are detected by examining the absorption spectrum of the coloured products formed, and are quant. removed by chromatography on a cation-exchange column (Dowex-50). The condensation with acetylacetone is carried out at 89° to 92°C; above 93°C the final coloured product shows a double-peaked absorption spectrum. Salt concn. and precision of neutralisation are critical. Optimum hydrolysis conditions are determined for each type of tissue studied with HCl concn. up to 6 N and for times up to 24 hr.

C. E. SEARLE

**341. Determination of glucuronic acid and glucuronates by means of the naphtharesorcinol reaction.** K. Heyns and C. Kelch (*Z. anal. Chem.*, 1953, **139** [5], 339-351).—The determination of glucuronic acid by naphtharesorcinol is described. The reagent is prepared by shaking vigorously 0.2 g of naphtharesorcinol with 100 ml of  $\text{H}_2\text{O}$ ; O is passed through the clear solution for 1 hr., and after setting aside for 24 hr at room temp., the excess of O is removed by passing N for 1 hr. Five ml of aq. test solution (glucuronic acid  $\approx 20$  to 150  $\mu\text{g}$ ) are treated with 5 ml of conc. HCl and 5 ml of reagent, then mixed thoroughly, and placed in a bath of boiling water for 30 min. The solution is cooled in ice-water for 10 min., extracted with 15 ml of ice-cold ether, and the ether solution is filtered through a pleated paper into a cylinder. When  $\approx 10$  ml have been collected, the cylinder is stoppered and kept in ice-water until the extinction is measured at 505  $m\mu$  by means of a suitable spectrophotometer. A calibration curve is prepared for the range 20 to 100  $\mu\text{g}$  of glucuronic acid, and a blank determination is made on 5 ml of  $\text{H}_2\text{O}$ .

R. E. STANTON

**342. Influence of cations on ninhydrin reaction for the determination of amino-acids.** Hanan Meyer and E. Riklis (*Nature*, 1953, **172**, 543).—In butanol 0.1 ml of 0.01 M  $\text{Al}^{+++}$ ,  $\text{Hg}^{++}$ ,  $\text{Fe}^{++}$ ,  $\text{Fe}^{+++}$ ,  $\text{Mn}^{++}$ ,  $\text{Sn}^{++}$ ,  $\text{Ag}^+$  or  $\text{Cu}^{++}$  completely inhibit colour formation between 10  $\mu\text{g}$  alanine and ninhydrin.  $\text{Ca}^{++}$ ,  $\text{Ba}^{++}$ , and  $\text{Cd}^{++}$  change the normal purple colour to a red. The actions of the cations can be inhibited by addition of the chelating agent versene (disodium ethylenediaminetetra-acetate). The method has been applied to the elution of spots from paper chromatograms.

H. G. BEVAN

**343. The chromatographic determination of amino-acids in the presence of large amounts of carbohydrate.** J. P. Dustin, C. Czajkowska, S. Moore and E. J. Bigwood (*Anal. Chim. Acta*, 1953, **9** [3], 256-262).—Mixtures of 15 common amino-acids (cystine, methionine and tryptophan excepted) are boiled with 6 N HCl in the presence or in the absence of carbohydrate. The decomposition products from starch and glucose do not interfere with the determination of the amino-acids on a column of Dowex-50 ion-exchange resin by a method previously described (*Anal. Abstr.*, 1954, **1**, 148). In no instance is the recovery of an amino-acid lowered by as much as 3 per cent. by addition of carbohydrate (2 g) to 25 to 50 mg of amino-acids per 200 ml of 6 N HCl.

W. C. JOHNSON

**344. An improved method of analysis for glycine using *Streptococcus faecalis*.** T. A. McCoy, M. K. Patterson, jun., and S. H. Wender (*Arch. Biochem. Biophys.*, 1953, **43** [2], 485-491).—*Streptococcus faecalis* A.T.C.C. 6057 is used satisfactorily to determine glycine in a final medium solution concn. of from 0.6  $\mu\text{g}/\text{ml}/\text{tube}$ . The growth response of the organism is measured by the turbidity of the suspension at the end of 16 hr. in a Fisher electro-photometer with a 650- $m\mu$  filter. Results of analyses of 7 purified proteins agree favourably with values of previous workers whilst the recoveries of glycine from protein hydrolysates (ranging in glycine content from 2 per cent. in casein to 43 per cent. in silk fibroin) are  $100 \pm 3$  per cent. The accuracy of the method, even for limited amounts of protein, is good.

D. BAILEY

**345. The determination of creatine substance with potassium mercuri-thiocyanate and dithizone. Part II.** P. Stelgens (*Biochem. Z.*, 1953, **324** [3], 228-236).—The procedure described in part I (*Brit. Abstr. C*, 1953, 359) for pure solutions is now extended to blood filtrates and urines. The results are compared with those of the picrate method of Folin, Lieb and Zacherl (*Hoppe-Seyl. Z.*, 1934, **223**, 169). The average creatine and creatinine content of blood is found to be 2.13 mg and 0.75 mg per cent., respectively. The new method is more specific and more sensitive. It is shown that heating urine on a bath of boiling water leads to losses in creatinine; treatment at 60°-65° C for 24 hr. is more satisfactory.

W. MANZ

**346. Separation and determination of ribonucleotides and related compounds by ionophoresis on filter paper.** W. C. Werkheiser and R. J. Winzler (*J. Biol. Chem.*, 1953, **204** [2], 971-981).—A detailed description is given of a method for separating purine and pyrimidine bases and their ribosides and ribonucleotides by ionophoresis on filter-paper strips. Ribonucleotides from < 1 mg of nucleic acids can be separated in high purity. The positions of the substances on the paper are found by observation under u.v. light or by passing the strips through

an adapted spectrophotometer. Quant. analysis is carried out by elution and measurement of the absorption at 260  $\mu$ ; aliquots are also analysed colorimetrically for phosphorus. C. E. SEARLE

**347. An antimony trichloride reagent suitable for the detection and estimation of non-ketonic steroids.** H. Rosenkrantz (*Arch. Biochem. Biophys.*, 1953, **44** [1], 1-8).—The effect is described of treating 51 steroids on paper with a conc. solution of  $\text{SbCl}_3$  in nitrobenzene followed by heating (5 min.) at 90° C. Observation of the characteristic colours shows that the procedure is satisfactory for detecting hydroxylated steroids and has a sensitivity of 1.5  $\mu\text{g}/\text{sq. cm.}$  The reagent is used to determine the relative mobilities in toluene-propylene glycol of 15 hydroxy steroids. These mobilities suggest that a number of hydroxy steroids can be separated in toluene-propylene glycol and this is confirmed by the resolution of a pure mixture of oestrol, oestrone and  $\alpha$ - and  $\beta$ -oestradiol in a 48-hr. chromatogram. D. BAILEY

**348. Reaction of steroid ketones with 2:4-dinitrophenylhydrazine [Determination of ketones as 2:4-dinitrophenylhydrazones].** H. Reich, K. F. Crane, and S. J. Sanfilippo (*J. Org. Chem.*, 1953, **18**, 822-832).—Methods are described for prep. and quant. isolation of 2:4-dinitrophenylhydrazones of steroid ketones. An excess of reagent is used, which is afterwards removed by oxidation with Benedict's reagent.

Two quant. methods are used to determine the amount of 2:4-dinitrophenylhydrazine utilised in the reaction. The first method involves conversion of the excess of reagent into acetone 2:4-dinitrophenylhydrazone, whilst the second involves a similar conversion into pyruvic acid 2:4-dinitrophenylhydrazone. The acetone deriv. is readily separated by chromatography whilst the acid deriv. is removed by extraction with aq.  $\text{Na}_2\text{CO}_3$ . The amounts of these deriv. are determined gravimetrically or spectrophotometrically; from the value so obtained, the no. of reactive carbonyl groups in an unidentified ketosteroid can be calculated. J. N. ASHLEY

**349. The determination of glutathione in the presence of nitrite.** R. A. Mortensen (*J. Biol. Chem.*, 1953, **203** [2], 855-859).—Nitrite interferes with the usual methods for reduced-glutathione determination. Modifications of the nitroprusside and amperometric methods in which sulphamic acid is used to eliminate the interference of  $\text{NO}_2^-$  are described. The necessary conditions for the removal of  $\text{NO}_2^-$  by sulphamic acid have been determined. The modified procedures have been shown to yield results in the presence of  $\text{NO}_2^-$  in satisfactory agreement with those obtained in its absence. B. VINEY

**350. A manometric assay for chymotrypsin.** R. E. Parks, jun., and G. W. E. Plaut (*J. Biol. Chem.*, 1953, **203** [2], 755-761).—A sensitive, easily performed, manometric assay for chymotrypsin has been developed which permits continuous activity measurements on many concurrent enzyme samples. The method is based on the esterase activity of chymotrypsin for certain amino-acid esters. The liberation of carboxyl groups during ester hydrolysis causes the evolution of  $\text{CO}_2$  from a  $\text{HCO}_3^-$  buffer. The rate of gas evolution, measured manometrically in the Warburg apparatus, is proportional to ester hydrolysis. L-Phenylalanine ethyl ester is the

most satisfactory substrate tested, being cheap, easily synthesised, and highly sol. in water. L-Tyrosine ethyl ester, N-acetylphenylalanine ethyl ester, and N-acetyltyrosine ethyl ester are more rapidly hydrolysed than is L-phenylalanine ethyl ester, but their usefulness as substrates is limited by the poor solubility in water of both the esters and their hydrolysis products, as well as by enzymic instability at the very low concn. of chymotrypsin that must be used with these substrates.

B. VINEY

**351. Synthetic dyestuffs and fermentation reactions. V. The influence upon catalase.** W. Diemair and K. Boekhoff (*Z. anal. Chem.*, 1953, **139** [4], 267-278).—The inhibiting effect of a series of dyes and their reduction products on the activity of catalase is studied by the volumetric measurement of  $\text{O}$  liberated from  $\text{H}_2\text{O}_2$  in a catalase-inhibitor- $\text{H}_2\text{O}_2$  system at pH 6.9. Azo, heterocyclic quinone, triphenylmethane dyes and nigrosin have no effect on enzyme activity. Of the azo dye reduction products examined,  $p\text{-C}_6\text{H}_4(\text{NH}_2)\cdot\text{N}(\text{CH}_3)_2$  and  $m\text{-C}_6\text{H}_4(\text{OH})_2$  cause 81 and 77 per cent. inhibition, respectively, whilst  $\text{C}_6\text{H}_5\text{NH}_2$ ,  $\alpha\text{-C}_{10}\text{H}_7\text{NH}_2$ ,  $m\text{-C}_6\text{H}_4(\text{NH}_2)(\text{OH})$ ,  $\alpha\text{-C}_{10}\text{H}_7\text{OH}$  and  $\beta\text{-C}_{10}\text{H}_7\text{OH}$  inhibit the action from 0 to 6 per cent.  $\text{C}_{10}\text{H}_8$  derivatives containing  $-\text{SO}_3\text{OH}$  and  $-\text{COOH}$  groups do not inhibit. The behaviour of these compounds is discussed in relation to their carcinogenic properties. C. G. TAYLOR

**352. Synthetic dyestuffs and fermentation reactions. VI. The effect on peroxidase.** W. Diemair and K. Boekhoff (*Z. anal. Chem.*, 1953, **139** [5], 352-359).—The influence of synthetic dyestuffs on the peroxidase activity of potato press juice has been investigated. The peroxidase activity is inhibited by concn. of rhodamine-B and of rhoduline orange-NO above 40 mg per litre, and acridine shows a similar effect. Rhodamine-B also arrests the diastase degradation of potato sarch. R. E. STANTON

**353. Correlation between percuprimetric titration curves of proteins and their constitution.** G. Beck (*Anal. Chim. Acta*, 1953, **9** [3], 241-248).—The curves obtained by a method previously described (*Brit. Abstr. C*, 1951, 387; 1952, 392, 396 and 497) are shown to give an indication of the nature of the constituent amino-acids. The biuret point and the turbidity point are also correlated with constitution. W. C. JOHNSON

**354. Estimation of zinc in biological material.** G. Weitzel and A.-M. Fretzdorff (*Hoppe-Seyl. Z.*, 1953, **292**, 212-221).—The method described is a modification of one of the dithione methods and is capable of estimating Zn from 1  $\mu\text{g}$  upwards. The sample is ashed with  $\text{H}_2\text{SO}_4$  and  $\text{H}_2\text{O}_2$  or  $\text{HNO}_3$ . The resulting solution is partly neutralised to pH 5.5 by the addition of alkali tartrate and NaOH and is extracted with diphenylthiocarbazone (I), in  $\text{CCl}_4$ . The zinc-(I) complex is ashed and dissolved in aq. tartrate and  $\text{Na}_2\text{SO}_4$ , and the Zn is determined polarographically. Provided the Zn-(I) complex is formed at pH 5-8, then  $\text{NH}_4^+$ ,  $\text{PO}_4^{3-}$  and Ca ions do not interfere. Copper, lead and cadmium are also detected by the method but are not determined quantitatively. L. ROWE

#### Drugs

**355. Spectrophotometric determination of barbiturates and the distribution of veronal (barbitone) in the organism.** I. S. Goldschmidt, W. Lamprecht and E. Helmreich (*Hoppe-Seyl. Z.*, 1953, **292**, 125-

137).—The usual method of determining barbiturates, the colorimetric measurement of the complex formed between barbiturates and cobalt salts at alkaline pH in the absence of water, cannot be used for the concentration ranges likely to be encountered within the body. The u.v. absorption of barbiturates in weak alkaline solution, max. approx. 255  $m\mu$ , min. 235  $m\mu$ , can be used for the estimation. As the position of the max. varies slightly for each barbiturate, a separate calibration has to be carried out for each substance. The tissues are extracted with chloroform and this solution is then shaken with 0.5 *N* NaOH. In this way background absorption is negligible. It is possible to determine down to 1  $\mu\text{g/ml}$  with an error of  $\pm 2$  per cent.

The distribution of barbitone in rat tissues was determined at set time intervals after lethal and sub-lethal doses of 120 mg and 30 mg, respectively, had been administered. The most marked accumulation of barbitone is found in the medulla oblongata of the brain. L. ROWE

356. The separation of alkaloids by paper partition chromatography. Part III. Assay of ergot. J. E. Carless (*J. Pharm. Pharmacol.*, 1953, 5 [11], 883-897).—The separation of the water-insoluble alkaloids, ergotamine, ergotamine, ergosine, ergotamine, and the water-soluble alkaloids ergometrine and ergometrinine by partition chromatography on buffered cellulose columns is described. The method has been successfully used to assay defatted ergot, only 0.5 g of sample being required and the assay being completed in 2 days. Recoveries of the pure alkaloids were consistently about 90 per cent., except for ergotamine of which about 80 per cent. was recovered. Comparison with the B.P. 1948 method was made by assays of 7 samples. The B.P. figures for total water-soluble alkaloids were considerably higher than those obtained by the chromatographic method in three samples, but reasonable agreement was attained in the remaining assays. N. M. WALLER

357. The estimation of tropane alkaloids in vegetable drugs by paper partition chromatography, with special reference to the alkaloids of *Datura sanguinea*. R. E. A. Drey and G. E. Foster (*J. Pharm. Pharmacol.*, 1953, 5 [11], 839-848).—The separation of tropane alkaloids on buffered paper (phosphate buffer pH 7.4) and the subsequent elution of the individual alkaloids is described. The recovered alkaloids are estimated by the Vitali-Morin reaction. The results in conjunction with the total alkaloid content may be used to determine the hyoscyne and hyoscyamine content of a sample. The assay has been applied to samples of *Datura sanguinea* and of *Duboisia* species. N. M. WALLER

358. Determination of alkaloids in tincture of *ipeacacuanha* by chromatographic adsorption. A. J. H. Schotman (*Pharm. Weekbl.*, 1953, 88 [45/46], 769-776).—Results obtained by adsorption on  $\text{Al}_2\text{O}_3$ , elution with 70 per cent. ethanol, and titration (after partial evaporation followed by dilution with water) of the eluate with 0.1 *N* HCl (*cf. Brit. Abstr. B.*, 1937, 1133) agree well with those obtained by the usual extraction methods. Any alkalinity contributed by the  $\text{Al}_2\text{O}_3$  is corrected for by a blank test, or, preferably, by previously washing out the column with 70 per cent. ethanol until the alkalinity found in the washings is caused only by traces of dissolved  $\text{Al}_2\text{O}_3$ . P. S. ARUP

359. The assay of tincture of digitalis and of the glycosides of *Digitalis purpurea*. H. Brindle, G. Rigby and S. N. Sharma (*J. Pharm. Pharmacol.*, 1953, 5 [11], 876-882).—An investigation of the potency of individual primary and secondary glycosides and the aglycones of *D. purpurea* has been made by the 18-hr. frog assay and a colorimetric assay based on the Kedde reaction with 3:5-dinitrobenzoic acid, or on the colour produced by heating with 33 per cent. HCl soln. for the glycosides. No correlation is observed between chemical and biological assays. The potency of digitalis is due mainly to the desacetyl-digilans and digitoxin. Gitoxin, gitoxigenin and digitoxigenin have a very low potency for frogs. The  $R_F$  values of several of the constituents of *D. purpurea* have been determined by paper partition chromatography and the constituents of 6 tinctures of digitalis have been investigated. N. M. WALLER

360. The B.P. and U.S.P. tests for pyrogens. J. G. Dare (*J. Pharm. Pharmacol.*, 1953, 5 [11], 898-910).—The relative efficiencies of the two tests and the sources of variation in the results are investigated. The max. sensitivity of both tests is attained when rabbits have not previously been given pyrogen, when the minimum restraint is imposed on the animal and when an electrical thermometer is used. It is suggested that these three requirements be added to the official methods. The U.S.P. method detects smaller amounts of pyrogen than does the B.P. test and it is suggested that the U.S.P. method of interpreting test data should replace the method used in the B.P. N. M. WALLER

361. Ascaridole studies. II. Iodimetric and polarographic methods of determination of ascaridole. A. H. Beckett and G. O. Jolliffe (*J. Pharm. Pharmacol.*, 1953, 5 [11], 869-875).—The discrepancies are investigated between the results obtained by the polarographic and the official (B.P. 1953) iodimetric method for the determination of ascaridole in oils of chenopodium. The differences arise because the B.P. factor is incorrect, and the amount of I used in this method is not directly proportional to the wt. of ascaridole added to the acidified KI solution. Titration figures from the B.P. method may be converted into correct ascaridole percentages by using the derived expression  $m = 0.00489n + 0.000275n^2$  where  $m$  = g of ascaridole reacting with the acidified KI soln. and  $n$  = ml of 0.1 *N*  $\text{Na}_2\text{S}_2\text{O}_3$  required (less blank titre). N. M. WALLER

362. Essential oil of the Spanish dictamnus. A. Portolés Alonso and J. Cabo Torres (*Inf. Quim. Anal.*, 1953, 7 [4], 113-128).—The oil content (max. found, 3.53 per cent. on dried heads) of samples from various locations, the distribution of oil in various parts of the plant (greatest in leaves) and the variation of oil content with stage of development of the plant (greatest at full floration) have been studied. Characteristics of the oil varied between the following limits:  $d_{40}^{20}$ , 0.9474-0.9619;  $[\alpha]_D^{20}$ ,  $+1.12^\circ$  to  $+5.08^\circ$ ;  $n_D^{20}$ , 1.5147-1.5166; m.p.,  $< -25^\circ\text{C}$ ; acid val., 1.6-1.9; sap. val., 8.7-9.0; I val., 96-103; hydroxyl val., 1.1;  $-\text{OCH}_3$  val. 15.6-15.8; b.p.,  $170^\circ\text{-}220^\circ\text{C}$ . The relative estragole (*p*-allylanisole) contents have been assessed from fractional distillation data. L. A. O'NEILL

363. Determination of penicillin G in penicillin O. J. L. Johnson, W. A. Struck, E. J. Scott and J. E. Stafford (*Anal. Chem.*, 1953, 25 [10], 1490-1492).—A method is described for accurately determining

small amounts (<1 per cent.) of penicillin G (benzylpenicillin) in penicillin O (allylmercaptomethylpenicillin). Oxidation of penicillin G with alkaline permanganate yields benzoic acid which is determined spectrophotometrically. Interfering oxidation products, which derive predominantly from penicillin O, are removed by extracting the benzoic acid with  $\text{CHCl}_3$  and transferring to aq. ammonia. Studies of known added increments of penicillin G have shown a recovery of  $86 \pm 4$  per cent. The method is directly applicable to potassium penicillin O, and a preliminary extraction of the organic base makes it applicable to the procaine or 2-chloro-procaine salt.

O. M. WHITTON

**364. The microbiological assay of mixtures of penicillin and dihydrostreptomycin.** D. G. Lewis and G. Sykes (*J. Pharm. Pharmacol.*, 1953, **5** [11], 933-937).—Methods for the analysis of dihydrostreptomycin and penicillin in mixed preparations are described. The assay of penicillin is dependent on the absence of interference, synergistic or otherwise, of the dihydrostreptomycin. It is carried out with *Staphylococcus aureus* and its reliability depends on the assay medium used, satisfactory results being obtained with a peptone-yeast extract. The assay of dihydrostreptomycin is carried out with *Bacillus subtilis* or *Bacterium coli*.

N. M. WALLER

**365. Chemical assay for neomycin.** J. D. Dutcher, N. Hosansky and J. H. Sherman (*Antibiot. & Chemother.*, 1953, **3**, 534-536).—A chemical assay procedure for neomycin B and C is described. It is based on the observation that neomycin B and C when heated with strong mineral acids yield furfural as one of the decomposition products. The quant. measurement of the furfural formed may be made in several ways, but the most direct procedure, when there are no interfering substances present, is by the measurement of the intensity of the absorption max. at 280  $\text{m}\mu$ . The method cannot be applied directly to fermentation broth because of the presence of interfering substances, but is of considerable use in following the purification of the neomycin concentrates.

I. JONES

**366. Cup-plate method for determination of erythromycin concentrations in serum and other body fluids.** A. Kirshbaum, F. W. Bowman, D. M. Wintermere and Eve R. Friedman (*Antibiot. & Chemother.*, 1953, **3**, 537-539).—A cup-plate agar diffusion method for determining the concentrations of erythromycin in serum and other body fluids is described. The method is a modification of the cup-plate diffusion assay of chloramphenicol and aureomycin (Randall *et al.*, *J. Clin. Invest.*, 1949, **28**, 940); it gives the actual concn. of the antibiotic within the inherent error of the biological method. Results of a comparative study between a serial dilution procedure and the cup-plate diffusion test are given; these show that the cup-plate method is more reliable.

I. JONES

**367. Determination of aureomycin in feeds by pad-plate method.** J. E. Grady and William L. Williams (*Antibiot. & Chemother.*, 1953, **3**, 158-164).—A pad-plate method for the assay of aureomycin, which is simple and can be adapted to 5 hr. or overnight determination, is described. The feed is extracted with a mixture of 4 N HCl, water and acetone (1 : 6 : 13 by vol.). Filter-paper pads are spotted with 0.1 ml of the extract, and then placed on an agar medium inoculated with *B. cereus*, and

incubated at 28°-30° C. The assay offers a method for studies of stability during various treatments of aureomycin supplemented feeds and correlation of aureomycin content with growth response. The method can be adapted to determination of aureomycin in milk, and, possibly, to the determination of aureomycin in blood. The sensitivity of the assay can be increased by decreasing the agar content of the medium, by refrigeration of assays to allow diffusion of the aureomycin into the surrounding medium, and by use of a single layer of agar medium.

I. JONES

## Food

**368. Colorimetric and fluorometric determination of vitamin A in fish liver oils; chromatographic separation of vitamin A and non-vitamin A materials in the unsaponifiable matter.** A. Fujita and M. Aoyama (*J. Biochem., Japan*, 1953, **40**, 157-168).—Two methods of estimating vitamin A are described, one indirect and the other direct. In the indirect method the total fluorescence value of the unsap. matter calcd. as vitamin A from about 10 mg oil are determined fluorometrically. The vitamin A is separated by chromatography in light petroleum on  $\approx 2.9$  g alumina, but in the 2 hr. taken by the separation some vitamin A is lost. The fluorescence value is therefore determined on the residue eluted with benzene and subtracted from the total value (see Fujita and Aoyama, (*Brit. Abstr. C*, 1952, 164). Hence the vitamin A is calcd. by difference. In the direct method the unsaponifiable matter from  $\approx 10$  mg oil is chromatographed on 0.8 g of weakly activated alumina and developed with light petroleum. The vitamin A is eluted with benzene and determined by known methods. The process takes 3 to 4 min. and no vitamin A is lost. The two methods agree well but no comparison is given with standard methods.

B. VINEY

**369. Determination of ascorbic acid by paper chromatography.** W. Heimann, R. Strohecker and F. Matt (*Z. Lebensmitt. Untersuch. u. Forsch.*, 1953, **97**, 263-270).—Ascorbic acid ( $R_F = 0.36$ ) is effectively separated from (possibly) accompanying reducing substances (cysteine, glutathione,  $\text{SO}_3^{2-}$ ,  $\text{S}_2\text{O}_3^{2-}$ ,  $\text{Fe}^{2+}$ ,  $\text{Sn}^{2+}$ , reducing sugars, etc.) by paper chromatography in an enclosed space by means of butanol-acetic acid-water (4 : 1 : 5) as ascending solvent (during 5.5-6 hr.). On spraying with  $\text{NH}_4$  molybdate solution [aq. 1.5 per cent.  $\text{NH}_4$  molybdate (3 ml) + 1 per cent. aq.  $\text{NH}_3$  (3 ml) + HCl-Na citrate buffer (Sørensen) (2 ml) + conc.  $\text{H}_2\text{SO}_4$  (3 drops)] a blue coloration is developed (the ascorbic acid is eluted by means of aq. oxalic acid and determined by dichlorophenolindophenol titration). The method detects 1  $\mu\text{g}$  of ascorbic acid, and can be used quant., but it does not admit of distinction between ascorbic and isoascorbic acids.

P. S. ARUP

**370. Determination of ascorbic acid by a new colorimetric reaction.** M. Schmall, C. W. Pifer and E. G. Wollish (*Anal. Chem.*, 1953, **25** [10], 1486-1490).—A new assay method described for ascorbic acid involves the reaction with diazotised 4-methoxy-2-nitroaniline in acid medium, and subsequent development of a blue colour in alkaline solution. This colour, with a maximum absorbancy at 570  $\text{m}\mu$ , is compared with standards in a suitable photo-electric colorimeter. The procedure is rapid, simple, and suitable for routine control. It can be

carried out directly, e.g., in the presence of dehydroascorbic acid and all other vitamins. Its sensitivity permits the determination of quantities down to 0.5 mg with a low limit of 10  $\mu$ g per ml, when a 50-ml sample aliquot is used. Precision is good and accuracy compares favourably with conventional procedures; a table of results shows a maximum deviation from the mean of 0.8 per cent.

O. M. WHITTON

**371. New fluorimetric method for the determination of ascorbic acid and mercaptan compounds.** H. Freytag (*Z. anal. Chem.*, 1953, **139** [4], 263-267).—Ascorbic acid, cystine, thioglycolic acid,  $\alpha$ -mercaptopropionic acid and Na mercaptobutane sulphate are determined by the fluorescence produced by the reduction of sodium 1:2-naphthoquinone-4-sulphonate (Folin's reagent) in u.v. light.

C. G. TAYLOR

**372. Quantitative estimation of ascorbic acid and related substances in biological extracts by separation on a paper chromatogram.** Yu-Tuan Chen, F. A. Isherwood and L. W. Mapson (*Biochem. J.*, 1953, **55** [5], 821-823).—L-Ascorbic acid and related compounds are separated on a paper chromatogram by the two solvent systems, *n*-butyl alcohol saturated with water and oxalic acid, and phenol saturated with water and oxalic acid. The phenolic solvent gives a clear-cut separation of L-ascorbic acid from D-araboascorbic acid. A quantitative procedure for the estimation of enediols from plant and animal tissues is described. Enzymes are inactivated by saturating the extract with oxalic acid, and the enediols are stabilised throughout subsequent operations by maintaining a high conc. of oxalic acid. The enediols are estimated after separation on a paper chromatogram by a colorimetric method with a solution of 2:6-dichlorophenolindophenol in 0.25 M Na<sub>2</sub>HPO<sub>4</sub>. Recovery of L-ascorbic acid and D-araboascorbic acid added to rats' urine and to extracts of cress seedlings was 85 per cent. even at the lowest conc. examined (0.5 mg/ml of urine or cress-seedling extract).

I. JONES

**373. Studies on the measurement of the folic acid and citrovorum factor potencies of certain natural products.** M. M. Andrews and B. S. Schweigert (*Arch. Biochem. Biophys.*, 1953, **44** [1], 165-175).—The pteroylglutamic (folic) acid and citrovorum factor potencies of yeast extract and animal tissues are estimated by differential microbiological analysis with *Lactobacillus casei* and *Leuconostoc citrovorum* 8081 as test organisms. The total folic acid activity of beef liver is increased considerably by the addition of cysteine during enzymatic or heat treatment, but little or no increases attributable to cysteine addition are observed with other animal tissues or with yeast extract. The effect of cysteine is more evident when *L. casei* is used than with *Streptococcus faecalis* R as the test organism. The most effective methods for the release of folic acid or citrovorum factor from bound forms are short-term autoclaving at pH 7, enzymatic treatment or autoclaving at pH 4, but these vary with different samples. With short-term autoclaving the total folic acid potency attributable to the citrovorum factor is approx. 10 per cent. for beef liver and kidney, 35 per cent. for yeast extract, 50 per cent. for ribeye muscle and 70 per cent. for beef spleen and heart. Thymidine is shown to contribute little to the total folic acid activity.

D. BAILEY

**374. The analysis of analogues. A general technique applied to the estimation of cyanocobalamin and hydroxycobalamin in mixtures of the vitamins.** J. G. Heathcote (*Chem. & Ind.*, 1953, [45], 1203). Partition between 2 immiscible solvents may separate two closely analogous substances. An aqueous solution of cyanocobalamin and hydroxycobalamin is shaken with benzyl alcohol. From a measurement of the optical density at 356 m $\mu$  in each separated phase the apparent distribution coefficient is evaluated. The coefficient is a function of the composition of the sample only and can be calculated from the partition coefficients of the pure substances.

MARGARET J. MARTIN

#### Sanitation

**375. Mass spectrometer determination of volatile contaminants in water.** F. W. Melpolder, C. W. Warfield and C. E. Headington (*Anal. Chem.*, 1953, **25** [10], 1453-1456).—A method is described for determining both qualitatively and quantitatively traces of volatile contaminants that affect the taste and odour of water. The volatile compounds are stripped from boiling H<sub>2</sub>O with H<sub>2</sub>, the vapour is condensed in a liq. nitrogen trap, and the condensate is analysed on the mass spectrometer. Petroleum products and other volatile compounds such as industrial solvents and cleaning fluids can be identified by this method, and gas and liquid hydrocarbons boiling up to at least 400° F in the concentration range of 0.01 to 100 p.p.m. can be determined in water.

O. M. WHITTON

#### Agriculture and Plant Biochemistry

**376. Determination of copper in plant material.** W. A. Forster (*Analyst*, 1953, **78**, 614-616). The disodium salt of ethylenediaminetetra-acetic acid (I) has been used to prevent interference from Al, Co, Fe, Ni, and Mn in the determination of Cu by means of sodium diethyldithiocarbamate (Sedivek *et al.*, *Brit. Abstr. C*, 1950, 513). By use of the ammonium salt of (I) with CCl<sub>4</sub> as extractant the method is considerably simplified and Al, Co, Fe, Mn, Ni, Zn, Ca, Mg and P cause no interference. The concn. of Cu in the CCl<sub>4</sub> extract is measured absorptometrically.

A. O. JONES

**377. Determination of calcium in plant material.** T. R. Williams and R. R. T. Morgan (*Chem. & Ind.*, 1953, [37], 970).—The material is ashed with HNO<sub>3</sub> and HClO<sub>4</sub>, the Ca is pptd. as oxalate, dissolved in HNO<sub>3</sub> and the solution is atomised in a flame photometer. The Ca content is read from a standard graph; the method is quick and accurate. The Ca contents of a number of leaves are given.

D. R. PECK

**378. Enzymic estimation of citric acid.** S. Dagley and E. A. Dawes (*Biochem. J.*, 1953, **55** [5], xxxv-xxxvii).—From anaerobic cultures of *Aerobacter aerogenes*, cell-free extracts are prepared that convert citric acid to pyruvate, acetate and CO<sub>2</sub>. Citric acid can thus be determined by incubation with the extract and estimation of the pyruvate formed by the method of Friedemann and Haugen (*J. Biol. Chem.*, 1943, **147**, 415).

C. E. SEARLE

**379. Retention of some flavins in paper chromatograms.** W. Forter and P. Karrer (*Helv. Chim. Acta*, 1953, **36** [6], 1530-1531).—The paper chromatography of flavins in butyl alcohol-formic acid-water media at 20° to 22° C on Whatman No. 1 filter-paper is described; the zones are examined by u.v.

fluorescence.  $R_F$  values are listed; they decrease when the number of  $-OH$  groups increases and the number of alkyl groups ( $CH_3$ -,  $C_2H_5$ -) decreases. The stereochemical structure of the sugar residues with the same number of C atoms has little effect on  $R_F$ , but hexose flavins show lower  $R_F$  values than corresponding pentose flavins. D. R. GLASSON

**380. The identification of rutin by paper chromatography, and its detection in tobacco extracts.** E. Wegner (*Z. anal. Chem.*, 1953, **138** [6], 423-426).—The direct identification of rutin in tobacco extracts by paper chromatography, as well as a method of identifying the quercetin and sugars produced on hydrolysis, is described. J. H. WATON

**381. New dispersion method for mechanical analysis of soils.** J. de la Rubia Pacheco and F. Blasco López-Rubio (*Inf. Quim. Anal.*, 1953, **7** [5], 143-148).—The sample is treated with  $NH_4$  acetate instead of HCl before sedimentation. This procedure is rapid and removes  $CaSO_4$  but not  $CaCO_3$ , the amount of which can be found in the silt and clay fractions. The "active  $CaCO_3$ " content of the soil (Dromineau, *Ann. Agron.*, Paris, 1932, 443) always lies between the amount of  $CaCO_3$  in the clay fraction and that in the clay and silt fractions combined, and is sometimes equal to the second of these quantities. The  $CaCO_3$  content of the clay fraction is a useful measure of the  $CaCO_3$  actively participating in the soil dynamics. The air-dried soil (20 g) is agitated with 15 per cent. aq.  $NH_4$  acetate (100 to 125 ml, with 3 to 5 ml of conc. aq.  $NH_3$  if the sample contains >10 per cent. of  $CaCO_3$ ) at just below the b.p. for 30 min.; it is filtered and washed with hot  $NH_4$  acetate solution (150 ml, or until free from  $SO_4^{2-}$ ) and then with boiling water until the washings are nearly colourless and salt-free. The sample is then passed through a 0.2-mm sieve, and after addition of 4 to 8 ml of  $N$  NaOH, it is sedimented by the International Method A.  $CaCO_3$  is determined in each fraction by any suitable method. If the soil contains >3 per cent. of org. matter it is pretreated with hot 12 per cent.  $H_2O_2$  (60 ml, repeated if necessary). D. P. YOUNG

**382. Fractionation of organic acids in sugar beets by ion exchange resins.** H. S. Owens, Alan E. Goodban and J. Benjamin Stark (*Anal. Chem.*, 1953, **25** [10], 1507-1511).—The background and details of a chromatographic method based upon the use of ion-exchange resins are given for the fractionation of organic acids in sugar beet diffusion juice. The method is satisfactory and may be useful for analysing other plant juices. The naturally occurring organic acids in beets appear in the following order: citric > oxalic > malic > glycollic. Pyrrolidonecarboxylic and lactic acid also appear in diffusion juices. The effect of processing, variety, and other agronomic factors on the organic acid content of sugar beets can now be studied. O. M. WHITTON

See also Abstracts 223, 229, 239, 243, 285, 291, 303, 307, 308, 311, 313, 314, 315, 319, 322, 323, 386, 391.

## 5.—GENERAL TECHNIQUE AND LABORATORY APPARATUS

### General

**383. A universal reagent for cleaning glassware.** R. H. A. Crawley (*Chem. & Ind.*, 1953, [45], 1205-1206).—A mixture of 5 per cent. HF, 33 per cent.  $HNO_3$ , 2 per cent. Teepol and 60 per cent. water is found to be a good reagent for removing obstinate

precipitates from glass and silica. It works by removing a thin layer of glass or silica and hence the nature of the deposit is immaterial; the reagent should not be used on graduated glassware.

MARGARET J. MARTIN

**384. Devices for continuous addition of solids and liquids on the laboratory scale.** D. E. Weiss (*Chem. & Ind.*, 1953, [29], 741-742).—**I. Liquids.** A cup, fitted with a U-siphon arm, is mechanically rocked in, and thus periodically filled from, a constant-level bath. As the cup is raised the liquid siphons out. The amount discharged can be altered by fitting the cup with a plunger. **II. Solids.** A vertical tube containing the solid is mounted non-coaxially about  $\frac{1}{4}$  inch above a rotating horizontal disc. The solid falls on to the disc and is blown from its path by a tangential air jet and then collected on a chute. G. LORD

**385. Preparation of thin tungsten fibres and their use in place of quartz.** J. O'M. Bockris and D. F. Parsons (*J. Sci. Instrum.*, 1953, **30** [9], 340).—A method is described of preparing uniform tungsten fibres of about  $3 \mu$  thickness by reduction of a 12-13- $\mu$  thoriated tungsten wire by a.c. electrolysis. These fibres are found to have a higher tensile strength and are considerably less brittle than quartz. J. E. H. KINGDON

**386. Simple automatic sampler for water.** R. Wilkinson and R. Briggs (*Chem. & Ind.*, 1953, [34], 886-887).—An apparatus is described for the automatic sampling of water at frequent intervals. The sampler, which is battery-operated, consists of a sample bottle fitted with a rubber stopper carrying two tubes, of which the inlet tube dips into the water to be sampled. The other tube is connected to an aspirator bottle slightly larger than the sample bottle; this bottle is mounted above the sample bottle and is filled with water. A siphon tube filled with water passes from the aspirator by way of a valve to a point of discharge at about the same level as the sampling point. The valve consists of a kink in a rubber tube retained by a short length of fuse wire connected between two metal pins fixed in insulating material. An electrical mechanism connected to a spring clock opens the valves at appropriate times on successive samplers by applying sufficient current to break the fuse; the rubber tubes then spring into a straight, siphoning position. The mechanism is illustrated. D. BAILEY

**387. A simple micromanometer.** E. Kovačič (*J. Sci. Instrum.*, 1953, **30** [9], 304-305).—Constructional details and mode of operation of an easily assembled micromanometer are given. The instrument works on the principle of the movement of an enclosed air bubble in a capillary tube. Because of the high magnification attained, pressure differences down to 0.01 mm of Hg can be measured. For differential air pressures water is used, and for differential water pressures a sealing liquid, immiscible with water, is used. J. E. H. KINGDON

**388. A simple apparatus for the calibration of hygrometers.** C. L. Cutting and A. C. Jason (*J. Sci. Instrum.*, 1953, **30** [9], 338-339).—Constructional and operational details of a simple apparatus for the calibration of hygrometers are given. The apparatus relies on a two-temp. method of maintaining a known relative humidity. J. E. H. KINGDON

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**389. Universal apparatus for continuous extraction of an aqueous solution with solvents more dense than water.** D. C. Ifland (*Anal. Chem.*, 1953, **25**, [10], 1577-1578).—The Wehrli extractor modified with a solvent return for continuous extraction of an aqueous solution with solvents (e.g.,  $\text{CH}_2\text{Cl}_2$ ) heavier than water is described and illustrated.

O. M. WHITTON

**390. Modified Hershberg - Wolf extractor.** E. Heftmann and David F. Johnson (*Anal. Chem.*, 1953, **25** [10], 1578).—In the extractor described and illustrated, which operates under reduced pressure, high-boiling solvents can be used for extracting heat-labile substances from aqueous solutions.

O. M. WHITTON

**391. Micro lipid extractor.** P. Mitchell (*Nature*, 1953, **172**, 124).—Apparatus for extraction of phospholipids by Reichert's method (*Helv. Chim. Acta*, 1944, **27**, 961) on a micro scale is described. The lower limit to the amount of material used in the original method was set by the loss accompanying the transfer to and from the extraction thimble. The proposed apparatus eliminates this transfer and the limit is determined solely by the gravimetric error. A diagram of the apparatus is given. Results are reproducible with 5 to 50 mg of material containing 0.3-5 mg of lipid when weighing is done on an air-damped aperiodic balance reading to 10  $\mu\text{g}$ .

G. W. CAMBRIDGE

**392. An apparatus for solvent-removal at low temperatures.** E. A. Bell (*Chem. & Ind.*, 1953, [29], 741).—The solution to be concentrated is trickled down a sloping lagged tube, up which warm air can flow. Indentations in the tube cause the air flow to be directed on to "lakes" of solution, thus increasing the rate of evaporation. G. LORD

**393. A variable temperature vapour jacket for fractionating columns.** J. F. A. Williams (*J. Sci. Instrum.*, 1953, **30** [9], 339).—A variable temperature vapour jacket for fractionating columns is described. It uses the constant temperature maintained in the vapour of a liquid boiling at a fixed pressure. With methyl benzoate temp. constant to  $\pm 0.25^\circ$  have been attained in the range  $45^\circ$  to  $193^\circ\text{C}$ , with pressures from 0.1 to 760 mm of Hg.

J. E. H. KINGDON

**394. Vacuum cold trap.** G. H. Miller (*Rev. Sci. Instrum.*, 1953, **24** [7], 549-550).—A cold trap is described in which the coolant is contained in an annular space surrounding the trap; it is useful where a collimated light or particle beam is present in a vac. system.

G. SKIRROW

**395. A modified Nesbitt absorption bottle.** C. F. Ellis and H. G. Durham (*Anal. Chem.*, 1953, **25** [9], 1430).—Caking of the Ascarite reagent, with consequent increase in the pressure drop around the bottle, is obviated by placing the upper portion of the column of Ascarite in a basket of glass tubing, which is removed after each determination.

J. M. JACOBS

**396. Analytical apparatus for determination of sulphur.** H. J. Kusa, Assr. to Standard Oil Co. (U.S. Pat. 2,634,360, 7.4.53—Appl., 21.9.50).—A hot tube leading to a combustion furnace contains a combustion boat. The electric heater for the tube is controlled by progressive increment to vaporize material in the boat; a reduction gear and const. speed motor are included for gradually switching on

the heating current. A switch is operated by the motor to substitute sequentially full line current directly to the heater by cutting out the controlling means.

O. POTTER

**397. Apparatus for automatically recording the absorption of oxygen.** J. C. E. Button and A. J. Davies (*J. Sci. Instrum.*, 1953, **30** [9], 307-310).—The design, construction and operation are described of an apparatus that records the mass of oxygen absorbed by a sample of material. This sample, contained in a 100-ml flask, reacts slowly with a thermostatically controlled constant-volume oxygen atmosphere, which is maintained at barometric pressure by replenishment with electrolytically generated oxygen. The electrolysis current is integrated electrolytically, producing a pressure proportional to the oxygen absorbed, which is recorded. The apparatus has no moving parts except those in the recorder, and a simple pressure operated switch. It is designed to be suitable for prolonged routine tests.

J. E. H. KINGDON

**398. Continuous oxidant recorder.** F. E. Littman and R. W. Benoliel (*Anal. Chem.*, 1953, **25** [10], 1480-1483).—To study the relationship between the occurrence of smog and fluctuations of the oxidant concentration in Los Angeles a continuous recorder that produces a direct reading record of oxidant concentration in terms of  $\text{O}_3$  equivalents is described and illustrated. It consists essentially of a continuous air-liquid contacting device and a recording colorimeter. Iodine is liberated from a buffered neutral KI solution by the oxidant and the light transmittance of the resulting yellow solution is measured by a double-beam colorimeter and continuously recorded on a strip chart. The contacting solution is regenerated by passing it through a bed of activated C. By maintaining constant air and liquid flows, the instrument can be calibrated directly in terms of  $\text{O}_3$ -equivalents in p.p.m. by volume.

O. M. WHITTON

**399. Temperature effects in a rotation viscometer.** C. C. Mill and E. R. Gates (*Anal. Chem.*, 1953, **25** [9], 1390-1393).—The heating effect in a rotation viscometer was investigated in connection with the study of the rheological properties of printing inks, when it is necessary to obtain torque versus rate of shear curves over a wide range of rates of shear. A rise of temp. occurs when the power input exceeds a certain crit. value and the heat developed at high shear rates modifies the curves. The experimental data can be corrected when the temp. coeff. of  $\eta$  of the material and the equilibrium temp. of the viscometer bob are known. The temp. rise of a sample during measurement in a rotary viscometer may be considerable and may account for the hysteresis loop found in flow curves by previous workers. For printing inks which have  $\eta$  of several hundred poises, the rate of temp. rise is likely to be appreciable.

J. M. JACOBS

**400. Fraction collector with continuously rotating turntable and improved receiver assemblies.** R. J. Dimler, J. W. Van Cleeve, E. M. Montgomery, L. R. Blair, F. J. Castle and J. A. Whitehead (*Anal. Chem.*, 1953, **25** [9], 1428-1430).—The apparatus is adapted to the collection of 2-4 litres of effluent in 10-50-ml fractions over a period of 8-24 hr. Elimination of the usual stepwise movement minimises the number of electrical and mechanical parts and thus the possibilities of failures of relays, timers, etc. The

conventional fragile distributor arm is also eliminated. Details are given of the turntables and their driving mechanism and of two types of distribution assembly. J. M. JACOBS

**401. Simple and inexpensive fraction collector.** W. J. Wingo and I. Browning (*Anal. Chem.*, 1953, **25** [9], 1426-1427).—The apparatus comprises a rotary distributing arm that is actuated by an indexing mechanism and a solenoid-controlled escapement. J. M. JACOBS

**402. Automatic fraction collector for chromatographic preparations.** J. L. Hickson and R. L. Whistler (*Anal. Chem.*, 1953, **25** [9], 1425-1426).—A collector for 25 to 250-ml fractions is described. The distributing arm is attached to the shaft of a 40-position stepping relay; by electrical impulses, at intervals of 2-55 min., timed by an automatic reset timer, this arm is moved from one receiver to the next. The funnels deliver alternately to the two concentric rows of receivers which may be 8-oz. bottles, thus permitting the collection of as much as 10 litres of effluent. J. M. JACOBS

**403. Fraction collector for chromatography.** E. Schram and E. J. Bigwood (*Anal. Chem.*, 1953, **25** [9], 1424).—The device comprises a siphon, which delivers a fraction of desired vol. (as little as 1 ml, with reproducibility better than 1 per cent.) each time the liquid reaches the upper elbow, a turntable with a notched disc actuated by a solenoid through a lever and spring pawl, and an electronic circuit in which the electrolytic contact between electrodes in the siphon (which occurs as the liquid rises to the proper level) modifies the grid potential of a "cut-off" triode. The amplified signal causes the relay controlling the solenoid to close. J. M. JACOBS

**404. Automatic volume fraction collector.** C. Mader and G. Mader, jun. (*Anal. Chem.*, 1953, **25** [9], 1423).—An inexpensive pipette switch is described which uses a siphon pipette similar to that of Riemann *et al.* (*Brit. Abstr. C*, 1952, 542) and delivers fraction volumes quantitatively. The pivot consists of a rubber stopper with copper wires inserted, to which the leads of a mercury switch are connected. The pivot wires balance in two slots covered with sheet copper from which connection is made to the collector mechanism. J. M. JACOBS

**405. An automatic fraction collector.** J. M. F. DeBroske and L. R. Crisp (*Rev. Sci. Instrum.*, 1953, **24** [7], 547-549).—The fraction collector described consists of a rotating table capable of holding 300 tubes. The table is driven by a synchronous motor and gearing that gives a rotation time between consecutive tubes of 1.6 sec. Overriding of the motor is prevented by an electric brake. Selection time is controlled by an automatic timer. G. SKIRROW

**406. Photoelectric volume-measuring accessory.** H. T. Dutton and F. J. Castle (*Anal. Chem.*, 1953, **25** [9], 1427-1428).—The device comprises a reservoir with a silvered-glass float which rises to interrupt a light beam and thereby closes a photoelectric relay starting the collector motor and beginning the movement of the next tube to collection position. Operation of the motor closes a switch which actuates a double-pole relay. One pole of the relay "latches in" the relay; the other pole operates a solenoid valve to drain the reservoir and start up a thermoswitch which finally unlatches the relay, whilst a cam switch stops the motor, lowers the float, and thus causes the photo-electric relay to open. J. M. JACOBS

### Optical

**407. Ultraviolet papyrography.** An apparatus for detecting substances on papergrams by ultraviolet light. Yohei Hashimoto (*Pharm. Bull.*, 1953, **1** [2], 176).—An apparatus is described in which paper chromatograms are irradiated with u.v. light of a definite wavelength and variations in the intensity of transmitted light are recorded automatically. By this means it is possible to detect unknown substances whose presence cannot be detected by existing methods. N. E.

**408. A new approach to direct reading spectrochemical analysis.** R. K. Brehm and V. A. Fassel (*J. Opt. Soc. Amer.*, 1953, **43** [10], 886-889).—A description of the design and performance of a new type of direct-reading instrument for spectrochemical analysis is given. A single multiplier phototube is used in combination with a rapid-scanning spectrometer. As the spectral lines cross the single exit slit, voltage pulses are generated by the phototube and are presented panoramically on an oscilloscope whose time-base is synchronised with the spectrum scan. The heights of the spectral line pulses are measured by converting them to bursts of constant amplitude pulses whose numbers are proportional to the magnitude of the original spectral line pulses. The method of conversion is described. The pulse bursts corresponding to the lines whose intensity is of interest are separated by means of electronic gates. A high-speed scaler is provided to reduce the count rate to a usable level. The total count registered is proportional to the time integral of the spectral line intensity. Intensity ratios are automatically computed after the exposure, and recorded on a variable span strip-chart recorder. Preliminary data indicate that a precision of analysis of  $\pm 1$  per cent. can be achieved. D. M. D. EGAN

**409. Precision of infra-red spectrometers in routine use.** E. Childers and G. W. Struthers (*Anal. Chem.*, 1953, **25** [9], 1311-1313).—Tests are described for the evaluation of the reproducibility and the linearity of the absorbance scales of single- and double-beam infra-red spectrometers. In routine infra-red analysis (the determination of cyclohexanone and cyclohexanol in cyclohexane) precision was evaluated from differences between the results of several individuals by the same procedure and instrument, and between those of two laboratories with different instruments. For duplicate determinations in the same laboratory the absolute expected error would be 0.05 to 0.08 per cent. J. M. JACOBS

**410. Double-beam densitometer and comparator.** R. O'B. Carpenter and John U. White (*Anal. Chem.*, 1953, **25** [10], 1473-1477).—A double-beam densitometer is described and illustrated; it has a servo-type automatic balancing system, a projection comparator, and a scale linear in density from 0 to 2.0. This is achieved by means of precision logarithmic aperture and a special electronic circuit, which provides a servo-loop gain, a stability, and a precision of  $\pm 0.003$  density unit, all of which are independent of the density being measured. O. M. WHITTON

**411. A new sensitive chemical actinometer. I. Some trials with potassium ferrioxalate.** C. A. Parker (*Proc. Roy. Soc., A*, 1953, **220**, 104-116).—Details are given of a new chemical actinometer of high sensitivity. The method depends on the

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spectrophotometric determination of the photolysis products of potassium ferrioxalate by means of the absorption of the ferrous compound of 1:10-phenanthroline.

The minimum energy detectable is  $5 \times 10^{-10}$  N quanta. Energies 100 times greater can be determined accurately to  $\pm 2$  per cent. The actinometer has been investigated in detail over the range 3650 Å to 3663 Å. Further tests show it to be suitable for use over a wide range of wavelengths, probably down to 2000 Å.

The approximate quantum efficiency has been determined at twelve wavelengths between 4900 and 2537 Å. The actinometer solution can be kept for long periods without decomposition and the photolysis products show very small decomposition in 24 hours even in air. The presence of oxygen causes little change in quantum efficiency at 3650 Å. The analytical procedure is rapid and simple. Stirring is unnecessary except for extremely precise work at least for longer wavelengths. The advantages of the method for the determination of small doses of radiation are discussed.

H. T. J. CHILTON

**412. Optical apparatus for inspecting the contour of an article.** Armstrong Siddeley Motors, Ltd. (Inventors: Jack Walker and P. Leng) (Brit. Pat. 695,822, 21.12.50).—The portion of the surface of the article being dealt with is brightly illuminated by means of high pressure discharge lamps. The light rays emanating from the illuminated portion pass through an objective lens to a screen on which a magnified image is thereby produced. The alignment of the arc is critically maintained, and in combination with it are two substantially parallel plates spaced to a predetermined extent and with the central plane of the space coinciding with that of the light sources. The plates have openings to receive the article, e.g., a turbine blade, of which the portion to be studied is in the central plane mentioned. The article is screened from all light except that passing through the plates; and cooling is achieved by a baffled inlet at the bottom of the casing and a similar exit at the top.

J. N. T. LINTOTT

#### Thermal

**413. A variable temperature and humidity oven.** J. N. Burcham (*J. Sci. Instrum.*, 1953, **30** [9], 335-336).—Air of a known humidity is produced by saturating air at a known temp. in a separate saturator and then heating the saturated air to the higher temp. of the oven. The R.H. is given by the ratio of the s.v.p. of water at the lower and higher temp. The construction and operation of both the saturator and oven are described. The R.H. may be set to any value between 30 and 95 per cent. over the temp. range 30° to 100° C.

J. E. H. KINGDON

**414. Apparatus for low temperature calorimetry.** R. W. Hill (*J. Sci. Instrum.*, 1953, **30** [9], 331-334).—The construction and full operating details are given of two pieces of apparatus for the measurement of thermal properties, particularly specific heat, of solid samples between 2° K and room temperature. The two machines differ in the method of using liquid helium, one incorporating a liquefier of the Simon expansion type, the other using liquid helium from a separate liquefier. The two methods of working are compared and their relative merits are evaluated. A general purpose calorimeter suitable for either apparatus is also described.

J. E. H. KINGDON

**415. Use of thermo-couples for measuring temperature below 70° K. A new type of low temperature thermo-couple.** T. M. Dauphinee, D. K. C. MacDonald and W. B. Pearson (*J. Sci. Instrum.*, 1953, **30** [ ], 399-400).—In view of the uncertainties in the measurement of low temp. ( $\approx 10^\circ$  K) with Cu-constantan thermocouples, the use of thermocouples composed of pure Cu against certain dil. Cu alloys (e.g., Cu + 0.005 per cent. Sn) for temp. in the range 2° to 30° K is advocated.

G. SKIRROW

**416. A new type of helium liquefier.** H. M. Long and F. E. Simon (*Nature*, 1953, **172**, 581).—An elastic metal bellows-type liquefier for He is briefly described. Liquid He is produced at a rate of 0.4 litre per hr. for a consumption of 1.2 litres of liquid air. Heat leaks and inefficient heat exchanging prevent the ultimate efficiency being reached. These defects are being remedied in a second design.

A. E. TRUSWELL

**417. Experimental ebullioscopic constants. Variation with molecular weight of solute and type of solvent.** C. A. Glover and Charlotte P. Hill (*Anal. Chem.*, 1953, **25** [9], 1379-1382).—By means of a simple ebulliometer and the differential thermometer described by Menzies (*J. Amer. Chem. Soc.*, 1921, **43**, 2309), the ebullioscopic constants were measured in several solvents commonly used for mol. wt. determinations, with solutes of various chemical types and mol. wt. of 110-891. In relatively non-polar solvents, e.g., benzene and *n*-heptane, the experimentally determined value of the ebullioscopic constant depends on the mol. wt. of the solute used in its determination, whereas in more polar solvents, e.g., acetone and ethyl alcohol this effect is not observed.

J. M. JACOBS

#### Electrical

**418. An electric micromanipulator.** V. Bush, W. R. Duryee and J. A. Hastings (*Rev. Sci. Instrum.*, 1953, **24** [7], 487-489).—The instrument contains four manipulator heads each mounting a microtool. The tool holders are suspended under spring loading by electrically-heated thermal expansion wires, these providing the fine control. Mechanical controls permit coarse adjustment.

G. SKIRROW

**419. A simple electrical method of recording small volume changes.** J. G. Davies (*J. Sci. Instrum.*, 1953, **30** [9], 306-307).—The recording pen of a float volume recorder has been replaced by an opaque flag which moves in a vertical plane between a light source and a photo-electric cell; the resulting electrical changes are recorded. Sensitivity can be varied by modifying the light intensity, by altering the photo-electric cell load resistance or by using a float recorder of different capacity; 1 mA/ml has been attained. Initial calibration of the instrument determines the range over which the output remains linear for given volume increments.

J. E. H. KINGDON

**420. Simple circuit for adapting thermocouple recorders to measure voltage in high-resistance circuits.** F. T. Gucker, jun. and Axel H. Peterson (*Anal. Chem.*, 1953, **25** [10], 1577).—A one-valve circuit is described which adapts a thermocouple recorder to an input impedance as high as 10,000 megohms without appreciable change in its voltage sensitivity or response characteristics.

O. M. WHITTON

**421. The electrolytic separation of elements at the mercury cathode.** R. Bock and K.-G. Hackstein (*Z. anal. Chem.*, 1953, **138** [5], 339-357).—A simple apparatus is described in which a large number of elements are separated electrolytically at the Hg cathode. Separation is favourably influenced by a large Hg surface and by stirring the soln. For all elements the lower the acid concn. the quicker and the more complete the separation. With acid concn. 0.1-6 N  $H_2SO_4$ , Cu, Zn, Cd, In, Tl, Sn, Bi and Fe, probably also Ag, Au, Hg, can be separated from solutions (1 g metal/100 ml soln.) in a max. of 8 hr. and with 99.99 per cent. efficiency. By the electrolysis of HF solutions quantitative separations of Fe and Ni are possible. R. J. MAGEE

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varying length, the number of pulses being proportional to the wave height. These operate one of a series of 32 P.O. meters, each corresponding to a particular wave height, and one count is recorded on the appropriate meter as the water level falls. The instrument is designed for laboratory use and field measurements. J. E. H. KINGDON

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See also Abstracts 232, 302.

# ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use in the text of an abbreviation or symbol the word is printed in full.

alternating current	a.c.	micro-litre	μl
ampere	amp.	micron	μ
Angstrom unit	Å	milliampere	mA
anhydrous	anhyd.	milligram	mg
approximate, -ly	approx.	millilitre	ml
aqueous	aq.	millimetre	mm
atmosphere, -ic	atm.	millivolt	mV
atomic	at.	minimum	min.
boiling-point	b.p.	minute (time)	min.
British thermal unit	B.Th.U.	molar (concentration)	M
calculated	(calc.)	molecule, -e, -ar	mol.
calorie (large)	kg-cal.	normal (concentration)	N
calorie (small)	g-cal.	number	no.
centimetre	cm	observed	(obs.)
coefficient	coeff.	organic	org.
concentrated	conc.	ounce	oz.
concentration	concn.	part	pt.
constant	const.	patent	pat.
corrected	(corr.)	parts per million	p.p.m.
critical	crit.	per cent. wt. in wt.	per cent. w/w
crystalline	} cryst.	per cent. wt. in vol.	per cent. w/v
crystallised		per cent. vol. in vol.	per cent. v/v
cubic	cu.	potential difference	p.d.
current density	c.d.	pound	lb
cycles per second	c.p.s.	precipitate	ppt.
decompos-ing, -ition	(decomp.)	precipitated	pptd.
density	ρ	precipitating	pptg.
density, relative	d or wt. per ml	precipitation	pptn.
derivative	deriv.	preparation	prep.
dilute	dil.	qualitative, -ly	qual.
direct current	d.c.	quantitative, -ly	quant.
distilled	dist.	recrystallised	recryst.
electromotive force	e.m.f.	refractive index	n <sub>D</sub> <sup>20</sup>
electron-volt	eV	relative humidity	R.H.
equivalent	equiv.	revolutions per minute	r.p.m.
experiment, -al	expt.	saponification value	sap. val.
gram	g	saturated calomel electrode	S.C.E.
gram-molecule	mole	second (time)	sec.
half-wave potential	E <sub>1/2</sub>	soluble	sol.
horse-power	h.p.	solution	soln.
hour	hr.	specific gravity	sp. gr.
hydrogen ion concentration	[H <sup>+</sup> ]	specific rotation	[α] <sub>D</sub> <sup>20</sup>
hydrogen ion exponent	pH	square centimetre	sq. cm
inch	in.	standard temperature and	
indefinite	indef.	pressure	s.t.p.
infra-red	i.r.	temperature	temp.
insoluble	insol.	ultra-violet	u.v.
kilogram	kg	vapour density	v.d.
kilovolt	kV	vapour pressure	v.p.
kilowatt	kW	volt	V
liquid	liq.	volume	vol.
maximum, -a	max.	watt	W
melting-point	m.p.	wavelength	λ
microgram	μg	weight	wt.

In addition the following symbols are used—

greater than	>	less than	<
not greater than	≥	not less than	≤
is proportional to	∝	of the order of, approximately	~

The principal Pharmacopoeias are denoted by B.P., U.S.P., or D.A.B., together with the identifying numeral.

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approximate, -ly	approx.	millilitre	ml
aqueous	aq.	millimetre	mm
atmospher-e, -ic	atm.	millivolt	mV
atomic	at.	minimum	min.
boiling-point	b.p.	minute (time)	min.
British thermal unit	B.Th.U.	molar (concentration)	M
calculated	(calc.)	molecul-e, -ar	mol.
calorie (large)	kg-cal.	normal (concentration)	N
calorie (small)	g-cal.	number	no.
centimetre	cm	observed	(obs.)
coefficient	coeff.	organic	org.
concentrated	conc.	ounce	oz.
concentration	conc.n.	part	pt.
constant	const.	patent	pat.
corrected	(corr.)	parts per million	p.p.m.
critical	crit.	per cent. wt. in wt.	per cent. w/w
crystalline	} cryst.	per cent. wt. in vol.	per cent. w/v
crystallised		per cent. vol. in vol.	per cent. v/v
cubic	cu.	potential difference	p.d.
current density	c.d.	pound	lb
cycles per second	c.p.s.	precipitate	ppt.
decompos-ing, -ition	(decomp.)	precipitated	pptd.
density	$\rho$	precipitating	pptg.
density, relative	$d$ or wt. per ml	precipitation	pptn.
derivative	deriv.	preparation	prep.
dilute	dil.	qualitative, -ly	qual.
direct current	d.c.	quantitative, -ly	quant.
distilled	dist.	recrystallised	recryst.
electromotive force	e.m.f.	refractive index	$n_D^{20}$
electron-volt	eV	relative humidity	R.H.
equivalent	equiv.	revolutions per minute	r.p.m.
experiment, -al	expt.	saponification value	sap. val.
gram	g	saturated calomel electrode	S.C.E.
gram-molecule	mole	second (time)	sec.
half-wave potential	$E_{1/2}$	soluble	sol.
horse-power	h.p.	solution	soln.
hour	hr.	specific gravity	sp. gr.
hydrogen ion concentration	[H <sup>+</sup> ]	specific rotation	$[\alpha]_D^{20}$
hydrogen ion exponent	pH	square centimetre	sq. cm
inch	in.	standard temperature and	
indefinite	indef.	pressure	s.t.p.
infra-red	i.r.	temperature	temp.
insoluble	insol.	ultra-violet	u.v.
kilogram	kg	vapour density	v.d.
kilovolt	kV	vapour pressure	v.p.
kilowatt	kW	volt	V
liquid	liq.	volume	vol.
maxim -um, -a	max.	watt	W
melting-point	m.p.	wavelength	$\lambda$
microgram	$\mu$ g	weight	wt.

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"The Simultaneous Determination of Cadmium and Magnesium with Disodium Ethylenediaminetetra-acetate," by E. G. Brown and T. J. Hayes.

The simultaneous determination of cadmium and magnesium by titration with a solution of disodium dihydrogen ethylenediaminetetra-acetate containing zinc sulphate is described. Selective control of the pH at 6.8 permits cadmium alone to be titrated, and magnesium is subsequently titrated at pH 10 in the same solution. Solochrome Black is used as indicator for both titrations. The molecular ratio of magnesium to cadmium must not be greater than unity for quantitative results, but a large excess of cadmium in the presence of magnesium can be satisfactorily determined. A theory is postulated for the reaction.

## NOTICES

### Vth International Spectroscopy Colloquium

UNDER the chairmanship of Prof. F. X. Mayer, the Spectrochemistry and Colorimetry Group of the Society of Austrian Chemists has undertaken the organisation of the next international Colloquium, which will be held at Gmunden, in Upper Austria, from August 30th to September 3rd, 1954.

As in previous Colloquia, both emission and molecular spectroscopy will be discussed and the following topics are proposed—

*Emission spectroscopy*—Analysis of non-conductors and of base metals, evaluation of spectrograms and experiences with methods not involving standard samples (*e.g.*, Harvey, Addink).

*Molecular spectroscopy*—Studies of artificial fibres, non-dispersive infra-red spectroscopy, Raman spectroscopy and a critical comparison of photo-electric and photographic methods of analysis.

As an innovation, it is proposed to devote the last hour of the afternoon session to correlated reviews of selected topics by one or more speakers relating their *personal* experiences. This will be followed by informal discussions, again stressing personal experiences.

Groups in each country have been asked to circulate invitations to their members and each country is asked not to submit more than five contributions. The final selection of papers and arrangement of the programme will rest with the Austrian Group. Comments on the Austrian proposals are invited, and those intending to take part in the Colloquium are requested to communicate as soon as possible with Mr. J. R. Stansfield, Hilger & Watts Ltd., 98, St. Pancras Way, Camden Road, London, N.W.1.

### Symposium on Analytical Chemistry, 1954

THE Midlands Society for Analytical Chemistry has made a final announcement on the Symposium on Analytical Chemistry being held at the University of Birmingham from August 25th to September 1st, 1954.

The programme includes original papers by 23 authors, lectures on recent advances in industrial application and special techniques by 17 lecturers, and three plenary lectures by speakers of international repute.

There will also be an exhibition of apparatus, reagents and literature, and new techniques will be demonstrated.

In addition to visits and social functions, including special visits for lady visitors, there will be a Library Exhibition of historical chemical literature, including the Joseph Priestley collection.

Registration forms and further information can be obtained from the Symposium Secretary, J. W. Robinson, B.Sc., Ph.D., A.R.I.C., 139, Stourport Road, Kidderminster, Worcs.

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